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WOOD PRODUCTS

DISTILLATES AND EXTRACTS

PART I.

THE CHEMICAL PRODUCTS OF WOOD DISTILLATION

PART II.

DYEING AND TANNING EXTRACTS FROM WOOD

BY

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GENERAL

A. I. L.

PREFACE TO THE FRENCH EDITION

Whilst perusing the sheets of the technical treatise which MM. P. Dumesny and J. Noyer have asked me to present to the public, I could not help being struck with the progress which chemical science has accomplished in the last thirty years—a progress which leads it, step by step, to explore all parts of the field of human activity, and to bring to each of those parts the measure of knowledge which it requires. At first the preoccupation of men of science was necessarily dominated by theoretical ideas, by verification of hypotheses, and by the building up of a system of doctrines capable of simultaneously satisfying not only the desire which we possess of demonstrating more and more intimately the constitution of matter, but also the need for simplification of the methods of teaching, and for the incessant realisation of fresh progress.

But realism never loses its rights. It was soon perceived that chemistry could not remain confined to the domain of the laboratory, that the problems of life were within its field, and that its scope became enlarged by association with the material works of mankind, by lessening human labour, and, consequently, increasing the riches and the happiness of different nations. Thus it is that we see men of science interest themselves to an ever-increasing extent in agricultural and industrial problems, and the teaching of industrial chemistry develop in all large countries, and so produce engineers capable of not only placing theory and practice in harmony in each industry, but capable also of modifying, of inventing and of sustaining the competition of products by diminishing their cost price.

It is with the same end in view that, alongside purely scientific literature, a need has made itself felt for technical literature, to

enlighten not only the young in their arduous search after knowledge, and not only the manufacturers themselves, but also their assistants, workmen and foremen whose professional skill is by this acquired knowledge relatively increased.

And here again a natural departure was made. It was to those industries which, working on a large scale and handling considerable capital, were—and should have been—able to be the first to make an appeal to the assistance of the man of science that attention was first of all directed. It is now the turn of industries which, occupying a secondary rank, are, nevertheless, of great importance, as much by the riches which they create as the necessity for the products which they place on the market, and *Wood Products, Distillates and Extracts* are of this latter category.

In France, a wooded country, the industrial chemistry of wood employs a great number of hands. The living which is made out of it is a somewhat hard one, since the charcoal which it prepares has to compete, especially on the markets of large towns, with many rivals, as a source of heat and particularly with gas. To maintain their ground, manufacturers have had, and will have, to improve their equipment continually, increase their range of bye-products, and increase the value of some of them, of the tar products especially; it is only by a complete knowledge of the properties of the raw material and its derivatives, and by the application of rational means of control to all parts of the factory, that they will be able to do so.

The industry of tannic extracts is of more recent date; the products which it prepares assume greater and greater importance, since the adoption of rapid methods of tanning has become a necessity in the hide and leather industry; this importance will continue to increase in proportion as chemical science is installed in the tannery in the place of the rule-of-thumb methods which have reigned there far too long. The preparation of these extracts should fulfil certain conditions; it requires an equipment which, to be conducted economically, must be continually supervised. And here again, as in the industry of the chemical derivatives of wood, it is the complete scientific knowledge of his trade which should guide the manufacturer, the foreman and the workman.

The treatise of MM. Dumesny and Noyer is, therefore, a timely one. Written by two practical men, distinguished engineers, who

know their subject, their collaboration could only result in the production of a book both scientific and technical, which contains all the instructions necessary for the methodical carrying on of each of the industries within its purview, and which consequently has its distinctive place in the factory laboratory and in the laboratory of the student. I have pleasure in presenting it to the public and to predict for it a deserved success.

E. FLEURENT,
Docteur ès Sciences,
Professeur de Chimie Industrielle au Conservatoire National des Arts et Métiers.

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TRANSLATOR'S PREFACE

THE translator has little to add to what Professor Fleurent has expressed so well. But the United Kingdom is far behind France in everything pertaining to woods and forests. We have no national school of forestry and no forestry inspectors. It is not to our national credit that our landed proprietors should be forced to recruit their foresters from amongst their most intelligent labourers without these having undergone any preliminary training or technical education to fit them for a situation of that nature beyond the facilities which the estate itself may afford for practical instruction. Again, notwithstanding the Laird of Dumbiedikes' instructions to his son, "Aye be stickin' in a tree, Jock ; it's growin' while ye're sleepin'," wide tracts of woodland are too often felled and not replanted, and the effects of sudden rainfalls are too often aggravated thereby in this country as well as in Corsica. But one step in the right direction has been taken by the Government quite recently, *viz.*, the purchase of a large tract of land as a model Highland estate, as an object-lesson to show how such an estate should be managed, with special reference to arboriculture. This new step on the part of the legislature augurs well for the future, but it would be well to put a special tax on unplanted land, and to relieve planted land of all taxes until it begins to pay for itself by thinnings, etc. Even a bounty would possibly not be out of place. One of the best object-lessons in arboriculture is to be seen on the Highland Railway between Perth and Inverness, where, thanks to the foresight of enlightened landed proprietors of the past, what would be a dreary tract of land is made to look in many spots like a paradise. Again, many European arboriculturists visit the famous oak forest of Altyre and Darnaway,

near Forres. The planting of these forests was, I believe, encouraged by a gold medal of the Society of Arts. But arboriculture does not now receive the encouragement it formerly did. The replacement of wooden ships by iron ships may account for the apathetic attitude of the legislature towards the development of British Forestry in the past.

Coming now to the utilisation of wood waste distillation. This has already been dealt with in a special treatise by E. Hubbard (Scott, Greenwood & Son, publishers). The difficulty of obtaining suitable retorts is discussed therein very fully. The sawdust from wood-paving blocks alone rises in London and other large towns into lofty accumulations. This should be distilled forthwith, and probably the revolving retort holds the key to the problem. The sawdust should not get waterlogged by exposure to rain when no retort can bring out of the decomposed sawdust what is not in it. The utilisation of bobbin waste in such thread manufacturing centres as Paisley should be highly remunerative. A recent series of articles, in the *Oil and Colour Trades Journal*, on American methods of wood distillation, with special reference to the distillation of wood for spirits of turpentine, may well be studied with profit. It cannot be too much urged on the attention of foresters and landed proprietors that both the Scots pine and the larch abound in turpentine, etc., and that the onus lies on them to extract it, and that it is as irrational to rear a forest of pines and larches without extracting the turpentine, etc., therefrom as it would be to keep a herd of famous dairy cows and not milk them. But we shall see what the Government will do with its own model estate. Spain alone exported 5,000 tons of turpentine and rosin, according to the last consular report. Now these 5,000 tons of valuable products were all taken from the air, not a particle from the soil. The trees which produce turpentine will grow in almost pure sand, and for timber alone, mixed pine and larch on such sandy soils will, after thirty years, yield a return averaging £1 per acre per annum from date of planting. That is on soil of no annual value otherwise! Such being the case, it may well be asked, Why have we any unoccupied so-called "barren" land? As to tanning extracts, our Australian cousins ought to encourage the culture of the wattle shrubs, and our Foreign Office ought to see to it that their cultivation in the Soudan is developed to the fullest extent. The extract

industry seems happily to be flourishing in Jamaica. What may be the ultimate effect of the freeing of grain alcohol in the U.S.A. for industrial purposes on the British wood-distilling industry remains to be seen.

DONALD GRANT.

LONDON, *June*, 1908.

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PART I

THE DISTILLATION OF WOOD

CHAPTER I

GENERALITIES

The industrial chemistry of wood—more especially the dry distillation of wood—has attained, for some years back, a considerable importance throughout the whole world. Thus in Europe alone the capital invested exceeds a milliard of francs (£40,000,000). On the other hand, the area annually occupied by the forests, which serve the pyroligneous industry, in certain countries, can be measured in hundreds of thousands of hectares (1 hectare = 2·47 acres), which annually produce hundreds of thousands of cubic metres of wood (1 cubic metre or stère = 35 cubic feet, 547 cubic inches, or 1·31 cubic yards). In North America alone they go so far as to fell whole forests every year, to manufacture, from the wood, the products exported into Europe by the United States. The industrial distillation of wood dates from 1798, the time of the experiments of Philip Lebon. Lebon, submitting wood to the action of heat in a retort, so as to decompose it, created the industry of wood carbonisation, which afterwards made immense progress at the time of the discovery of the coal-tar colours, the manufacture of which required large quantities of acetic acid and methylic alcohol. A new outlet for methylic alcohol was created later on, when the present laws, within a short interval, regulated in France and in Germany the use of wood-spirit for denaturing industrial alcohol. In France are felled every year 250,000 to 300,000 stères of oak, beech, etc.;[1] that enormous amount of timber being out of proportion to the extent of the annual plantation in French forests tends to a prejudicial deafforestation of the soil, which will be readverted to in the chapter dealing with the extraction of tannic acid from the chestnut tree. Nevertheless, the French consumption of methyl alcohol greatly exceeds the French production, but the French exports of acetic acid, as the following statistics for 1903 and 1904 show, greatly exceed the French imports thereof.

[1] A stère being 35 cubic feet and a cord 125 cubic feet, to bring stères to cords multiply by $\frac{7}{25}$.

TABLE I.—SHOWING IMPORTS AND EXPORTS OF METHYL ALCOHOL INTO AND OUT OF FRANCE DURING YEARS 1903 AND 1904

French Imports from (1903)	Metric Tons.	French Exports to (1903)	Metric Tons.
Germany	232·501	Switzerland	3·761
Belgium	347·824	Other foreign countries	0·514
Other foreign countries	189·839	Free zone	8·786
		Algeria	5·340
		Other colonies and protectorates	1·792
Net weight	770·164	Net weight	20·193
Value	£32,346·88	Value	£852·12

1904: French imports, gross weight, 1,712·8 metric tons, value £50,000. 1904: French exports, gross weight, 43·1 metric tons, value £1,240.

TABLE II.—SHOWING IMPORTS AND EXPORTS OF ACETIC ACID INTO AND OUT OF FRANCE DURING YEAR 1903

French Imports from	Metric Tons.	French Exports to	Metric Tons.
Germany	1·077	Great Britain	15·862
Belgium	3·355	Switzerland	1·306
Other foreign countries	·294	Spain	25·340
		Italy	13·846
		Turkey	47·460
		Other foreign countries	36·751
		Algeria	15·523
		Tunis	7·239
		Other colonies and protectorates	4·791
Net weight	4·726	Net weight	168·118
Value	£47·28	Value	£3,362·36

1904: French imports, gross weight, 1·6 metric tons. 1904: French exports, gross weight, 236·9 metric tons, value £3,960.

On the other hand, Germany shows the greatest production, owing to the development of the chemical industries, which use up large quantities of acetic acid. Thus in 1897 Germany treated 400,000 stères of dry wood, which produced 1,500 to 2,000 metric tons of wood-spirit, 8,000 to 10,000 metric tons of acetate of lime, 10,000 metric tons of wood tar and 44,000 metric tons of wood charcoal. Since 1880 America has exported large quantities of wood-spirit and acetate of lime to European countries. This exportation has assumed considerable importance during the last ten years. The greater number of American installations, which may be estimated at about a hundred and are syndicated, delivered into Europe in 1901 2,500 tons of wood-spirit, the product of 200,000 tons of dry wood.

Up to now wood-spirit is the only chemical product which combines within itself all the properties required in the denaturation of alcohol so as to render it unfit for consumption (undrinkable). In France the proportion of denaturant added to alcohol, being 10 per cent. of methyl alcohol containing 25 per cent. of acetone, used up in 1901 more than 44,000 Imperial British gallons of wood-spirit, the value of which exceeded £80,000. On the other hand, Germany, which employs its denaturants in smaller doses, uses up a still larger amount; thus, with such industries as the manufacture of colouring principles, aniline and methyl aniline, special varnishes, etc., its consumption in 1901 was 26,400,000 British Imperial gallons, of which 1,100,000 gallons were for heating apparatus. During that year the quantity of methylic alcohol, used by Germany in its different industries, represented a value of about £1,600,000 in spite of its low selling price, which was twenty-four to twenty-eight shillings per 22 gallons. On the other hand, the consumption of acetic acid in the colour industry was greater than that of methylic alcohol.

Varieties of Wood used in the Wood Charcoal Industry.—All sorts of wood may be used in wood distilling; however, according to the yield and the products desired, preference is given to one or other variety, and that according to their density and their resistance to strain. Wood may for this purpose be classified thus: *Very hard,* Hawthorn; *Hard,* Maple, Box and Wild Cherry; *Rather hard,* Oak, Plum, Elm; *Slightly hard,* Beech, Hazelnut, Pear, Chestnut; *Soft,* Pine, Spruce, Larch, Birch, Alder, Horse Chestnut; *Very soft,* Limes, Poplars and the different varieties of Willows. Ordinary woods give more acetic acid than alcohol; woods of the spruce-fir kind yield more tar. The amount of charcoal is about the same for both kinds of wood.

TABLE III.—AMOUNT OF PRODUCTS YIELDED PER CORD OF WOOD.[1]

Class of Wood.	Char. Bush.	Alcohol. Galls.	Calcium Acetate. Lbs.	Tar. Galls.	Wood Oils. Galls.	Turpentine. Galls.
Hard Woods . .	40-50	8-12	150-200	8-20	—	—
Resinous Woods .	25-40	2-4	50-100	30-60	30-60	12-25[2]
Sawdust . . .	25-35	2-4	45-75	—	—	2-10[3] —

Chemical Phenomena connected with the Carbonisation of Wood.—The results obtained are quite different according to whether the wood is distilled slowly or rapidly. Thus Violette found that elder wood which yielded 18·9 per cent. of its weight of charcoal by slow distillation only gave 9 per cent. by quick distillation.

[1] Veitch, U.S.A. Dep. Agric. [2] Light wood.
[3] Sawdust. (*This Table is added by Translator.*)

TABLE IIIa.—SHOWING THE RESULTS OBTAINED BY SLOW (A) AND QUICK (B) DISTILLATION OF WOOD (SENFFT)

	Charcoal.		Gas.		Distilled Products.		Pyroligneous Acid.		Acetic Acid.	
	A.	B.	A.	B.	A.	B.	A.	B.	A.	B.
Hornbeam .	25·37	20·47	22·23	31·01	52·04	48·52	47·65	42·97	6·43	5·23
Alder . .	31·56	21·11	17·91	31·13	50·53	47·76	44·14	40·70	5·77	4·13
Birch . .	29·24	21·46	19·71	35·56	51·05	42·98	45·59	39·74	5·63	4·43
Rowan .	27·84	20·20	20·62	33·40	51·54	46·40	44·11	39·99	5·56	4·16
Beech . .	26·69	21·90	21·66	33·75	51·65	44·35	45·80	39·45	5·21	3·86
Aspen .	25·47	21·33	27·09	32·31	47·44	46·36	40·54	39·45	5·10	4·36
Oak . .	34·68	27·73	17·17	27·03	48·15	45·24	44·45	42·04	4·08	3·44
Fir . .	34·30	24·24	18·78	29·41	46·92	46·35	40·99	40·11	2·30	1·78
Larch .	26·74	26·06	21·65	32·17	51·61	43·77	42·31	38·19	2·69	2·06

These results explain themselves, if it be borne in mind that, in bringing wood quickly to a high temperature, the carbonic acid formed, and the water vaporised, become dissociated in contact with the red-hot charcoal, the first yielding carbonic oxide by absorption of an amount of carbon equal to that which it already contains, the second yielding carbonic oxide and hydrocarbides by combination (1) of its oxygen with carbon, (2) of its hydrogen, likewise, with carbon. For that same reason, air-dried wood should be preferred to wood freshly felled, or to wood transported by river floating. Such woods contain a rather considerable amount of water capable of reducing the yield of charcoal. The amount of carbon transformed depends also on the temperature of the carbonisation, as was first demonstrated by Pettenkoffer's experiments, and also by the following results obtained by Violette upon 100 parts of wood previously dried at 150° C. :—

TABLE IV.—SHOWING THE EFFECT ON THE CARBON CONTENT OF THE CHARCOAL OF CARBONISING WOOD (PREVIOUSLY DRIED AT 150° C.) AT DIFFERENT TEMPERATURES (VIOLETTE)

Temperature to which Wood was brought.		Residuum or Charcoal obtained.	Carbon per cent. in Residuum.	Temperature to which Wood was brought.		Residuum or Charcoal obtained.	Carbon per cent. in Residuum.
C.	°F.			°C.	°F.		
150	302	100·00	47·50	290	554	34·10	72·50
160	320	98·00	47·60	300	572	33·60	73·23
170	338	94·60	47·77	310	590	32·90	73·63
180	356	88·60	48·93	320	608	32·25	73·57
190	374	82·00	50·60	330	626	31·75	73·55
200	392	77·10	51·80	340	644	31·50	75·20
210	410	73·15	53·37	350	662	29·65	76·64
220	428	67·50	54·57	432	809·6	18·90	81·97
230	446	55·40	57·14	1023	1873·4	18·75	83·29
240	464	50·80	61·30	1110	2030	18·40	88·14
250	482	49·60	65·88	1250	2282	17·95	89·10
260	500	40·25	67·80	1300	2372	17·45	90·70
270	518	37·15	70·45	1500	2732	17·31	94·56
280	536	36·15	71·64				

Summing up, the woods most suitable for carbonisation, so as to obtain acetic acid and a good charcoal, are hard woods, such as oak, beech and hornbeam. They should not be felled before they are about twenty years old, and they should be felled preferably in winter, so that the wood may not be full of sap.

Products of the Destructive Distillation of Wood: (1) *Gaseous.*—Carbonic oxide and carbonic acid. (2) *Pyroligneous.*—Hydrocarbides, methyl alcohol, crotonylic and amylic alcohols, ether, acetone, formaldehyde, methylol, acetic, propionic, butyric and valeric acids, and, finally, nitrogenous compounds of an ammoniacal type, amine and pyridine. (3) *Tars.*—Hydrocarbides (benzenes and paraphenes), then once more methylic alcohol and acetic acid, higher fatty acids, monophenols and diphenols, a little pyrogallol, dimethylic ether and homopyrogallol. (4) *Residuum.*—Wood charcoal.

Properties of Principal Products of the Distillation of Wood: (1) *Wood Charcoal.*—Good charcoal shows the grain of the wood that produced it; it is black and sonorous (that is, it emits a clinking sound when struck), it is not easily crushed, and does not soil the fingers. It floats on water in virtue of the numerous pores that it contains. It is a bad conductor of heat and electricity. Wood charcoal burns without a great flame; it burns more readily the lower the temperature at which it was produced. Thus charcoal made at 350° C. (662° F.) takes fire almost suddenly, whilst that made towards 1,500° C. (2,732° F.) is only kindled with some difficulty. Wood charcoal has a density which varies somewhat with the wood that produced it. On an average, the hectolitre (2¾ bushels) weighs 14 to 18 kilogrammes (11 to 14½ lb. per bushel) in the case of charcoal from soft woods; 22 to 28 kilogrammes (17½ to 22 lb. per bushel) in the case of charcoal from hard woods; and 30 to 35 kilogrammes (22 to 28 lb. per bushel) in the case of oak charcoal.

TABLE V.—SHOWING THE COMPOSITION OF THE THREE TYPES OF WOOD CHARCOAL

Kind of Wood.	C	H	O	N
Soft wood	85·18	2·88	3·44	2·46
Hard wood	87·43	3·26	0·54	1·56
Oak	88·20	2·80	7·40	1·60

The calorific intensity of wood charcoal is generally comprised between 6,500 and 7,000 calories. By carbonisation in closed vessels the density of the wood charcoal so obtained is very feeble, and its calorific intensity is less high than by forest carbonisation.

(2) *Methyl Alcohol.*—Methyl alcohol (CH_3OH) is a mobile liquid with a decided spirituous odour; it boils at 66° C. (150·8° F.); its density is 0·814 at zero. It mixes with water in all proportions. The

density at 15·5° C. of methylic alcohol diluted with water has been determined by Ure.

TABLE VI.—SHOWING THE DENSITY OF METHYL ALCOHOL OF VARIOUS STRENGTHS (URE).[1]

Alcohol per cent.	Density.	Alcohol per cent.	Density.
100	0·8316	60	0·922
90	0·843	50	0·941
80	0·872	40	0·956
70	0·899		

Methylic alcohol is separated from its aqueous solution by carbonate of potash. It burns with a pale, scarcely luminous flame, and oxidises in presence of platinum black, with production of formic aldehyde. Finally, methylic alcohol exercises a peculiar physiological action on the organism, which renders its use in food or drink very dangerous. A stère of wood (35 cubic feet) yields on distillation 2 to 3 litres of alcohol. [For American yield of alcohol per cord, see Table III., page 3.]

(3) *Creosote.*—Creosote is not a well-defined product; it consists of phenol, accompanied by several of its higher homologues: cresyl hydrate or cresylol, and, according to Berthelot, of phlorol, cresol, and especially gaïacol. Creosote is a colourless, oleaginous liquid, which turns yellowish in the light; its density is 1·037 at 20° C. (68° F.); it boils at 203° C. (397·4° F.). Slightly soluble in water, creosote dissolves very well in alcohol, ether, fixed and essential oils, acetic acid and alkaline lyes. Heated with caustic soda and manganese dioxide, it forms a product which, taken up with water, yields rosolate of soda, from which sulphuric acid precipitates rosolic acid, a colour much used in yellow dyeing or calico printing.

(4) *Acetic Acid.*—Acetic acid (CH_3CO_2H) is a colourless liquid with a density 1·064. It crystallises below 17° C., boils at 120° C. (248° F.). Its odour is suffocating, but, diluted with water, it is rather pleasant. Applied to the skin, acetic acid destroys the epidermis and produces vesication. It attracts moisture from the air, and mixes in all proportions with water, and with alcohol mixed with water; up to a certain proportion, this acid increases in density, to diminish afterwards, when the proportion of water is exceeded. The maximum density of dilute acetic acid is 1·073. It then corresponds to a mixture of 77·2 of pure acid and 22·8 per cent. of water, and although this is not a definite hydrate it is expressed by the formula $C_2H_4O_2,H_2O$. The vapour of

[1] A diluted alcohol taken as standard (100 per cent.). See Dittmar and Fawsitt's Table in Appendix.—D. G.

acetic acid is inflammable and burns with a blue flame; the glacial acid has the property of not decomposing carbonate of lime. It must be diluted with water before it reacts on that substance; a mixture of alcohol and acetic acid does not redden litmus and does not attack certain carbonates (Pelouze).

(5) *Acetone* (C_3H_6O).—Acetone is a colourless inflammable liquid burning with a luminous flame; it is volatile, its odour is ethereal, its taste burning; its density is 0·792 at 18° C. (64·4° F.) and 0·814 at 0° C. (32° F.). Its boiling point is 56° C. (132·8° F.). Acetone does not solidify at 15° C. (59° F.). Soluble in all proportions in water, alcohol and ether, acetone neither dissolves calcium chloride nor potassium chloride, but it dissolves the greater number of the resins, oils and fats, camphors, etc. Gun cotton is easily soluble therein. Acetone forms, with alkaline bisulphites, crystallisable compounds, insoluble in an excess of bisulphite as well as in ether, but soluble in boiling alcohol, from which they crystallise on cooling. These compounds are likewise soluble in water and easily decomposed on boiling with an alkaline carbonate or an acid. Under many circumstances it is thus possible to isolate acetone or to purify it by causing it to enter into this state of combination. As concentrated a solution of bisulphite as possible must be used. The reaction then takes place more rapidly and with disengagement of heat.

TABLE APPENDED TO CHAPTER I.—SPECIFIC GRAVITY AND WEIGHT PER CUBIC FOOT AND PER CORD OF DIFFERENT KINDS OF WOOD.[1]

	Density.	Ash.	Lbs. per Cubic Foot.	Lbs. per Cord.
Ash	0·625-0·7184	0·26-0·78	38·9-46·8	2790-3350
Oak	0·6540-0·9501	0·26-1·49	40·7-59·2	2920-4220
Chestnut	0·4504	0·18	28·0	2010
Beech	0·6883	0·51	42·9	3070
Birch	0·5760-0·6533	0·25-0·31	35·9-40·8	2570-2920
Poplar	0·3889	0·96	24·2	1740
Elm	0·6506	0·80	40·5	1910
Longleaf Pine	0·6999	02·5	43·6	3120
Norway Pine	0·4854	0·27	30·2	2160
White Pine	0·3854	0·19	24·0	1720
Spruce	0·4051-0·4584	0·27-0·3?	25·2-28·6	1810-2050
Hemlock	0·4239	0·46	26·4	1890
Douglas Fir	0·5157	0·08	32·1	2300
Larch	0·6236	0·33	38·8	2780
Maple	0·5269-0·6912	0·33-0·54	32·8-43·1	2300-3090

[1] Sharple's Tenth Report U.S.A. Census. (*This Table is added by Translator.*)

CHAPTER II

PRINCIPAL METHODS OF CARBONISING OR "COALING" WOOD

THE different systems of carbonising wood may be grouped under one of the two following heads: carbonisation (1) by partial combustion; (2) without access of air. The first category includes the methods used in the forest, simply to convert the wood into charcoal; such charcoal is recognised as being more valuable than that got by one of the processes of the second category, where it is rather a by-product, unless it be quite a special product, such as charcoal for gunpowder.

Carbonisation by Partial Combustion.—These processes may be subdivided into two sections: (1) carbonising in stacks or the forest process; (2) carbonising in ovens for the production of tar.

(1) *Carbonising in Stacks or the Forest Process.*—Generally, to reduce freight charges, wood is "coaled" in the forest where felled. As the wood must, above all, be protected from the action of the air, stacks are constructed in which the carbonisation is produced at the expense of the wood to be carbonised. On a dry soil, protected from the wind, a suitable site is prepared called the *foulde*. The soil chosen should neither be light nor sandy. Neither should it be compact, so that it may readily absorb the liquids which condense during carbonisation. When possible, a *foulde* which has already done duty is again utilised; the yield in charcoal is then better, and may amount to 20 per cent. The floor ought to slope from the centre to the circumference with a circular drain, so that carbonisation may be more easily effected. Only one and the same variety of timber should be carbonised in the same stack. The stack is built thus: Around some billets of wood, placed vertically in the centre of the *foulde*, chumps of wood 10 inches long are arranged so as to stand upright in layers, packing them as tightly as possible the one against the other. The diameter of each layer—separated by a few pieces of wood lying flat—gradually decreasing, imparts to the stack the form of a hemisphere or a paraboloid, the height of which is 10 to 13 feet. When the stack is finished it is covered by a layer of small plants, moss or leaves, then with a layer of earth or turf, leaving apertures or vents in the lower part. Then

the upright billets are removed from the centre, which thereby leaves a vacant space, forming a central chimney, communicating with the vents in the lower part of the stack. The chimney is then filled with small pieces of lighted wood, which set fire to the mass, and, when combustion is sufficiently far advanced, the chimney, which up to now has been well fed with fuel, is stopped up; then, starting from the top, the vents are opened so as to allow the products of combustion to escape. The smoke which first escapes is black, but it soon becomes transparent; when it is bright blue, which shows that the carbonisation is in the vicinity of the vents, the latter are stopped up, and others are opened, 10 or 12 inches lower down, and so on. When the bottom is reached all the apertures are closed, and the stack is covered with a layer of moist earth. It is now allowed to cool for several days; the earth is then removed and the charcoal produced is separated from the imperfectly carbonised portions which are termed *fumerons*. Oak and beech thus yield 72 to 75 per cent. by volume (21 to 23 per cent. by weight) of wood charcoal. Hornbeam yields 55 to 60 by volume, and 21 per cent. by weight. For the carbonisation of resinous wood in Sweden and in Austria the stacks, instead of being vertical, are horizontal. They have the advantage of carbonising a large quantity of wood, whilst entailing less labour. The carbonisation is, moreover, more easily managed, and the charcoal obtained more uniform in quality.

(2) *Carbonisation in Ovens or Kilns for the Production of Tar.*—The use of ovens, generally, entails building expenses, and afterwards involves the transport, always costly, of the raw materials, chiefly wood.

Rectangular Ovens.—The use of these ovens is a transition between the stack process and that of ovens properly so called. They are built to remain stationary and consist of a sort of rectangular brick kiln which may be closed hermetically. Vents to regulate the progress of the operation are placed at the four corners of the rectangular kiln. From 200 to 250 stères of wood are piled in this chamber, making flues at the same time which lead to the vents. The fire is brought to the centre of the oven, through a hearth situated in its axis, and carbonisation goes on, from within outwards, by means of the hot gases. The tar and pyroligneous acid collect at the bottom of the apparatus. It has also been attempted to make circular ovens on the same principle, but they have been abandoned, because they yielded wood charcoal of a quality inferior to that produced by the stack process.

Chabeaussière's Oven.—The object of this oven was to collect the by-products of wood distillation in addition to wood charcoal. Carbonisation is effected in pits built of masonry, 10 feet deep, 10 feet

wide at the bottom, and about 11½ feet at the top. These pits are closed by a dome-shaped, wrought-iron lid, pierced, first of all, by a central hole for lighting the wood, then by four other holes used to watch the extinction of the charcoal. Vents bring the air necessary for the combustion, and serve also for regulating the draught. The billets are piled horizontally in the oven, care being taken to leave a chimney in the centre. The wood is then lit, and all the apertures in the lid are stopped and the lid itself covered by a layer of earth. The distillation lasts four days.

Schwartz Oven.—The principle of these ovens is based on the fact that a flame which does not contain free oxygen cannot burn wood, although it can decompose it. In this class of oven the carbonisation chamber has the shape of an elongated rectangle covered by an arch;

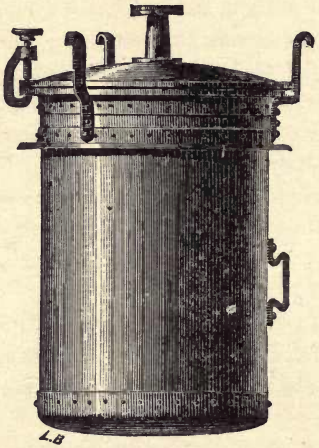

FIG. 1.—Portable vertical retort for wood distillation.

on each of the two long sides of the oven the flues from an outside fireplace convey a flame without oxygen, which brings the wood to a temperature sufficiently high to carbonise it. The gaseous products escape by conduits in the middle of the small sides of the oven, and the liquids, which condense in the bottom, are collected by siphons which lead them into receivers in the vicinity.

Chinese Ovens.—In China the ovens are underground, and the chimney ends in the bottom of the oven; another opening on the ground level serves to regulate the entrance of air into the newly lit oven, so as to produce slow combustion, which is modified according to the colour of the smoke, which escapes by the chimney.

Carbonisation in Closed Vessels: Portable Retorts; Fixed Retorts; Continuous Retorts. (1) *Portable Retorts.*—One of the oldest of these, still used

in France, consists of a well-riveted, vertical, cylindrical, wrought-iron boiler, 10 to 14 mm. thick (say from $\frac{2}{5}$ to rather over $\frac{1}{2}$ inch), 2 to 2·3 metres deep (say from $6\frac{1}{2}$ to $7\frac{1}{2}$ feet), and 1·5 metres in diameter (say 5 feet). It is closed on the top by a lid luted with clay, and fixed by iron catches or by screw bolts. The lid has a central pipe, for the disengagement of the vapours. To the top part of the retort, underneath the circular flange, on which the lid rests, there are fixed three bent pieces of iron, by which it can easily be hooked under a crane, so as to be readily deposited in, or brought out of, the furnace. An iron handle, fixed a little less than half-way up, facilitates the tilting of the retort, when, after distillation, it is desired to empty out the charcoal which it contains. Finally, the retort rests on the iron crown of the furnace by a strong flange, riveted on to the cylinder, about 40 centimetres (say $15\frac{1}{2}$ inches) lower down than the top flange of the retort. The life of these retorts is from five to six years for the bottoms, and ten to twelve years for the cylindrical bodies. These cylinders are arranged in series of 10, 16, and even 24, in two rows, in the same mass of masonry, built so that the flames from each of them do not play on the iron, which might become overheated. The bottoms of the retorts are, therefore, protected by fireclay bridges. The furnace gases circulate round the cylinder by flues made in the masonry arch so as to escape to the chimney, by an upper orifice placed directly underneath the iron crown of the furnace. A furnace consumes 200 kilogrammes of coal (say 4 cwt.) in winter, and 150 kilogrammes (say 3 cwt.) in summer per twenty-four hours in carbonising 10 stères of wood, say on an average 15 to 20 kilogrammes per stère (cubic metre), say 33 to 44 lb. of coal. When the importance of the installation admits of it, the retorts are sometimes heated by gasogenes adjacent to the bench of retorts, so as to avoid the loss of heat inevitable with long conduits. The handling of the retorts is done by the aid of a double rolling bridge, carrying an elevating apparatus or hoisting tackle both wrought by hand or electrically as shown in Fig. 2. The retort, when distillation is complete, is withdrawn from the furnace, and transported outside, to be emptied of the charcoal which it contains, whilst another retort, charged with wood, is put in its place, so that the work suffers no interruption. When the retort containing the charcoal is sufficiently cool, it is opened, then it is placed on a bascule to empty it of charcoal. In certain factories, to diminish the number of retorts and consequently the initial installation capital, the cylinder is opened as soon as it comes out of the furnace house, so as to tilt its glowing contents into wrought-iron extinguishers. Advantage is then taken of the retort being in a horizontal position to charge it with fresh

wood. Sometimes a sort of iron crate is used to receive the retort from the crane, so as to facilitate emptying; these crates have two diametrically opposite pivots around which they can turn. An adjustment fixes the retorts in the crates. In some installations a single

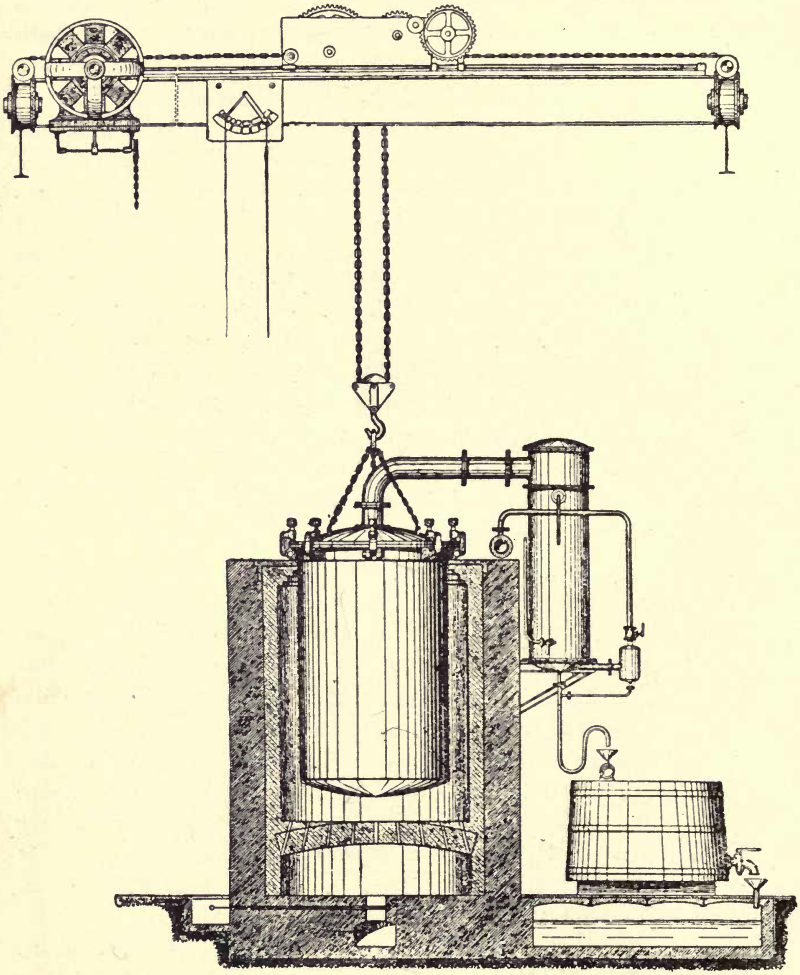

FIG. 2.—Portable vertical retort for wood distillation with hoisting and lowering tackle.

hearth does duty for two carbonisation furnaces. By means of dampers for the furnace gases, and of small fans for the permanent gases, these gases are directed into one or other of the furnaces, so as to bring the retort, which is nearing the end of its distillation, to a higher tempera-

ture. By this arrangement a constant fire can be kept on the grate and a considerable saving in fuel realised, as well as great regularity in working, which does away with the *fumerons* (half-burnt pieces) in the charcoal.

(2) *Fixed Retorts—Vertical and Horizontal—and Large-sized Continuous Retorts.* A. *Cylindrical Vertical Retorts.*—This type, being almost obsolete, will only be described as a matter of history. Mollerat frères were, in 1810, in France, the first to invent this type of plant for extracting pyroligneous acid from wood heated in closed vessels. Their apparatus consisted of a large wrought-iron cylinder of 3 cubic metres capacity, built into a brick furnace, heated by a fireplace under the retort, and so constructed that the combustion gases circulated round the cylinder before passing to the chimney. A manhole was inserted in the lid for charging the cylinder and for the discharge of the charcoal from the carbonisation of the wood, when the operation was terminated. A pipe on the side of the upper part of the retort led the distillate to a condenser. Kestner afterwards modified this plant by closing the cylinder by a removable lid held by iron clasps, and inserting an orifice in the bottom for the discharge of the charcoal, which was then collected in cast-iron extinguishers. This system entails comparatively a much larger plant to work the same quantity of wood than an installation with portable retorts.

B. *Horizontal Cylindrical Retorts.*—In Great Britain, in Sweden and in Germany, wood is distilled in cylinders laid horizontally in the furnace and heated by the same fireplace (Fig. 3). The two ends are closed either by clay-luted, cast-iron doors or by cast-iron doors with two handles, and kept in position by a system of iron crosspieces, carrying a tightening screw like gas retorts. One end of the cylinder has in its closing disc, which is not dismantled except for repairs, a pipe for the disengagement of distilled products. As to the other door, it may be used for charging the wood into the retort, and then for discharging the charcoal, which is run into wrought-iron extinguishers. These cylinders take a charge of 5 stères of wood, the distillation of which takes eight hours. This type of retort has lately undergone many modifications. The retort has now only one door, as shown in Fig. 4, placed in the smoke-box of the furnace. This plan gives better results in the carbonisation of the wood, as all parts of the retort are heated. A charge of up to 5 stères of wood takes sixteen to twenty-four hours.

C. *Large-sized Furnaces.*—In certain German districts large horizontal retorts are used, capable of taking a charge of 30 to 50 stères of wood. They are heated, not only on the exterior, but also on the interior

by two wrought-iron chimneys starting from the bottom of the retort. A charge in this type of retort takes four to six days.

The *Stack Furnaces,* a view of which is given in Fig. 6, are on the same plan as those used in America, in Russia and in Hungary. They are built of masonry and—everything else being equal, even as regards

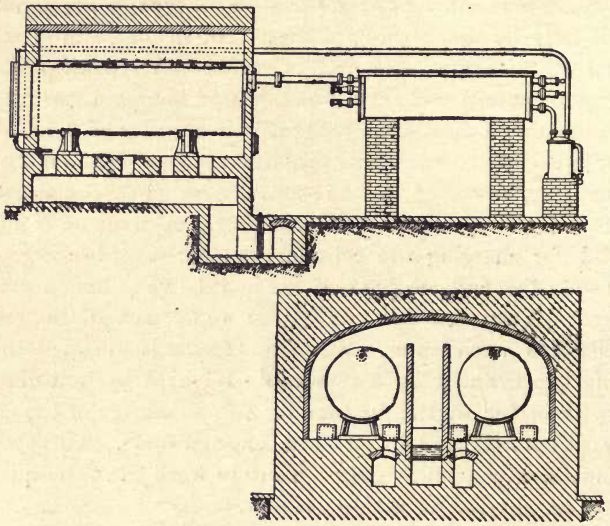

Fig. 3.—Cross section and longitudinal section of furnace or oven with two horizontal retorts for wood distillation and arrangement for utilising the gas produced for heating the retorts.

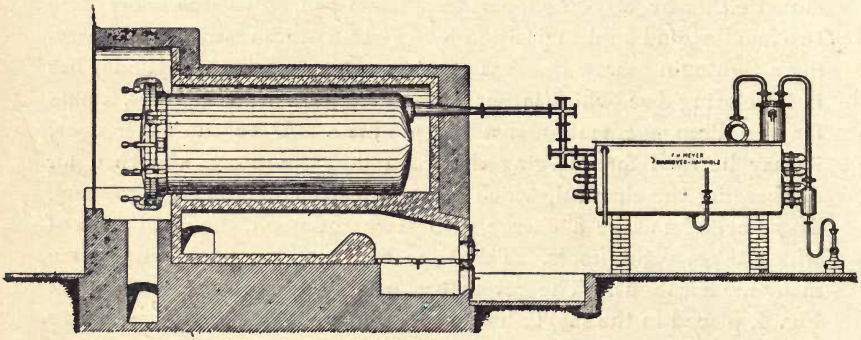

Fig. 4.—Furnace for wood distillation with two horizontal retorts with only one door for closing.

products obtained—run, in proportion to size, cheaper than other plant. They can be charged up to 300 stères (84 cords), each charge taking twenty days. At the exit of the condensers the gases are propelled by a fan under the stacks, where they are burnt as fuel.

D. *Continuous Distillation Plant. The Astley Paston Price Furnace.*—This furnace, built of fireclay bricks, is a large flue, the floor of which slopes slightly. It can take three trucks charged with wood. It is closed at the ends by iron doors, and is divided by wrought-iron partitions separating the middle truck from the two others. The fireplace being directly underneath the second truck, the latter is then in the hottest part of the furnace and is in full distillation whilst the first is cooling,

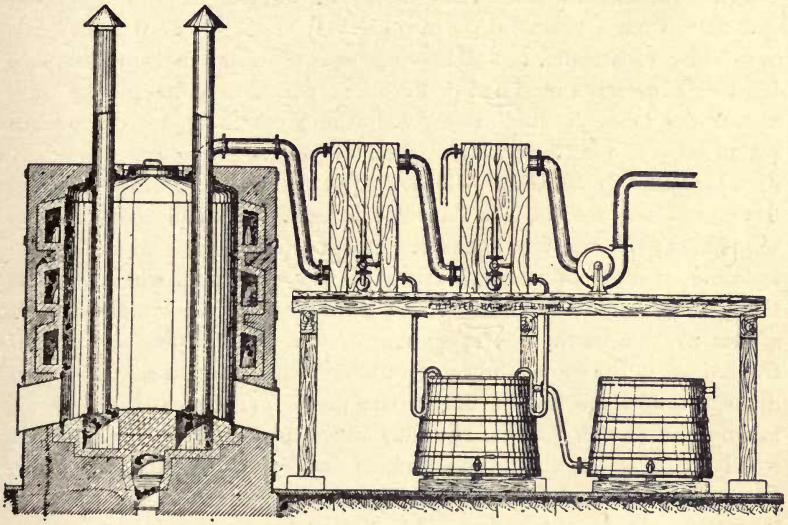

FIG. 5.—Capacious vertical retorts for wood distillation with internal and external heating arrangements.

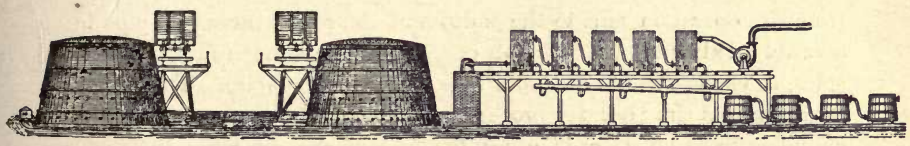

FIG. 6.—Masonry stack ovens or furnaces for wood distillation on very large scale and appropriate condensation plant.

protected from air, and the third, the last introduced, is being preliminarily dried. The condensable gases escape by pipes fixed in the roof of the middle compartment.

Bresson's Vertical Furnace is a derivative of the preceding, but it differs in the plant used and in the fact that the wood to be carbonised is shifted vertically instead of horizontally. It consists of five superimposed, jacketed, cast-iron cylinders, supported by a piston in the bottom. The three middle cylinders are in full heat in a masonry

furnace whilst the lower cylinder is cooling and the wood in the top one is undergoing preliminary drying. Horizontal dampers allow the three cylinders in course of distillation to be isolated. The volatile products disengaged from the wood escape through a pipe, which starts near the uppermost part of the third upper cylinder and leads to a condenser, whilst the permanent gases go to a gasometer, from which starts a pipe which brings them back to the fireplace, where they are burnt. In this furnace wood distils in forty-eight hours, passing gradually from a temperature of 250° to 700° C. (482° to 1,292° F.). According to Bresson, the yields in pyroligneous products are notably higher than those obtained with other plant, in consequence of a slower and more methodical carbonisation; moreover, the charcoal so produced is of a better quality.

Distillation of Sawdust.[1]—This style of distillation could be carried out in ordinary retorts, but it would present some difficulties, in consequence of the moisture contained in the sawdust, etc. Moreover, an isolating envelope of charcoal forms in the beginning, which renders the penetration of the heat to the centre of the mass difficult, and afterwards retards the disengagement of the distilled products. Confronted with the impossibility of rationally distilling these waste products, resource has been made to the use of mechanical methods for mixing the raw material. Holliday invented a plant in which the wood waste is fed in at one end of the retort through a hopper, then, whilst an Archimedean screw gradually leads it to the other extremity, the material is carbonised in its passage through the apparatus; the gases and vapours disengaged are led by a special pipe to a condenser, whilst the very finely divided wood charcoal which results from this operation falls to the bottom of the retort through a pipe in a vessel filled with water. This process consumes much fuel, and, besides, it yields a dilute distillate very costly to evaporate. To remedy these defects the Holliday process was modified by the use of several cylinders fixed above each other, the bottom one receiving the main heat from the fire, whilst the top one is heated by the waste heat. The wood waste is introduced in the top part of the plant, where it is freed from the bulk of its water. It then describes a zig-zag course as it passes from one cylinder to another, to issue completely carbonised through the bottom of the lower cylinder. In another process, retorts analogous to an acetone retort are used. Two similar retorts, one above the other, communicate by pipes. In the top retort a preliminary drying is effected, in the second the distillation proper.

[1] For a full treatment of this branch of wood distilling, see Hubbard's *Utilisation of Wood Waste* (Scott, Greenwood & Son).

Meyer's plant for the distillation of wood waste consists of two horizontal retorts, into which there are introduced, through two sliding doors, trucks with a certain number of shelves, on which the material to be distilled is spread in a thin layer. Whilst carbonisation is going on in the one retort, the wood waste is being dried in the second. The carbonised wood waste is withdrawn on the trucks into cooling chambers. By this method, owing to mixing being done away with, working expenses are less than in the previous methods, and finally the hot retorts can be recharged immediately. Bergmann tried, whilst still using ordinary retorts, as his patent shows, to agglomerate wood sawdust in the form of briquettes, and thus avoid the drawbacks already referred to; but this process was soon abandoned, as, in order to distil these compressed materials, the retorts must be brought to a high temperature, and the plant wore out too quickly.

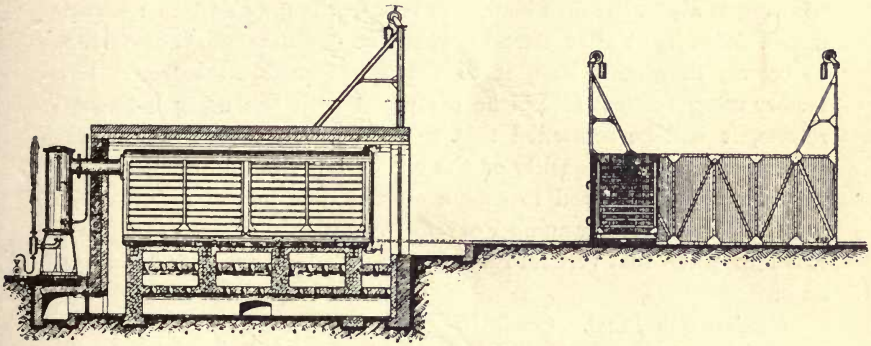

FIG. 7.—Retort for the carbonisation of smalls or wood waste.

FIG. 7A.—Retort coolers for cooling the retorts (Fig. 7) after an operation.

Working Routine : (1) *Horizontal Retorts.*—The wood charcoal having been just discharged, the retort is filled with wood as dry as possible, charging the bottom first; the door is closed and the fire urged. In ten minutes the distillation commences, as is seen by the great increase of temperature in the goose-neck; then it distils condensable products, accompanied by permanent gases. The liquid, which condenses first of all and is run away because it contains the greater part of the hygroscopic water contained in the wood, is slightly acid, and holds in solution some traces of tar. Then the strength in acid increases, and the colouration, owing to the greater amount of tar, is deeper. For retorts of 2 cubic metres capacity the distillation lasts about fourteen hours; at the end of that time the quantity of condensed products diminishes, the disengagement of gas ceases, and when the goose-neck begins to cool the operation is finished. The fire is with-

drawn and the door of the cylinder is soon opened, and the wood charcoal, which then becomes incandescent, is withdrawn by means of rakes. It falls into wrought-iron receivers, in which it cools without access of air. The extinguishers are then taken away, and after making sure that the neck of the retort is not obstructed by condensed tar, a fresh charge is made with wood brought on a truck.

(2) *Vertical Retorts.*—The cylinder being filled with 4 or 5 stères of wood, taking care to utilise its capacity to the utmost, its lid is put on, and it is laid in its place in the furnace; distillation commences; it is carried on open to the air until all moisture is expelled, that is to say, until the previous white smoke becomes blue. The neck of the retort is now connected with the disengagement tube, which is luted with clay. So long as the distillate does not run clear it is collected apart to be converted into brown acetate of lime; when it becomes bright brown and tests strong (*de titre fort*), which occurs in normal working at about the fifth hour, it is sent to the grey acetate reservoir direct. Ten hours after starting, when the distillation jets commence to brown, they must be sent to the brown acetate reservoir. If a factory using vertical retorts be compared with one using horizontal retorts, it will be remarked that there is no advantage in the first system, except in the quality of the charcoal produced, provided that this charcoal be allowed to cool a certain time in the retort, which involves a much greater number of retorts. Everything else being equal, it is recognised that vertical retorts have no advantage over horizontal ones.

Condensation Plant. Condensers.—As in every similar industry, the condensation of the distillate in wood carbonisation plays a great part. A badly planned installation may lead to mishaps, which can only be prejudicial to the manufacturer. In fact, the gas from the retort brings in its train acetic acid, acetone and wood-spirit, and, as wood yields about 20 per cent. of its weight of permanent gas, it follows that in the case of a factory treating 50 tons of wood a day, 8,000 cubic metres of gas are produced, capable of bringing in their train pyroligneous products, which are burnt in the fire. In summer, as the temperature of the water used for condensation is higher, as much as 50 per cent. of methyl alcohol and acetone and 10 per cent. of acetic acid may be lost. It is therefore necessary in installing a factory to impart to the refrigerating condensers a rather large surface and development, so that, even with the strongest distillation of gas and vapours, the distilled products are brought to the temperature of cold water as they issue from the condenser. No hesitation should be made in installing complete and sometimes costly condensation plant, especially when it is

a case of powerful carbonising plant. As tarry deposits are apt to form in the pipe which carries away the distilled products, which, by becoming calcined, would obstruct the pipes, it is well not to have the condensers too far away from the retorts. In the early days of wood distilling the condenser consisted simply of four horizontal copper pipes supported on a wooden or iron frame, communicating with each other by three elbow joints, and completely enveloped by wrought-iron jackets, in which water circulated. This cold water flowed from a reservoir into the lowest jacket, close to where the condensed products issued, then passed successively into the other upper jackets by means of small vertical pipes, which connected the annular spaces between the pipes and their jackets. The condensed products flowed into a reservoir, whilst the permanent gases were led by special piping to the retort furnaces, where they produced in burning such a heat that for the

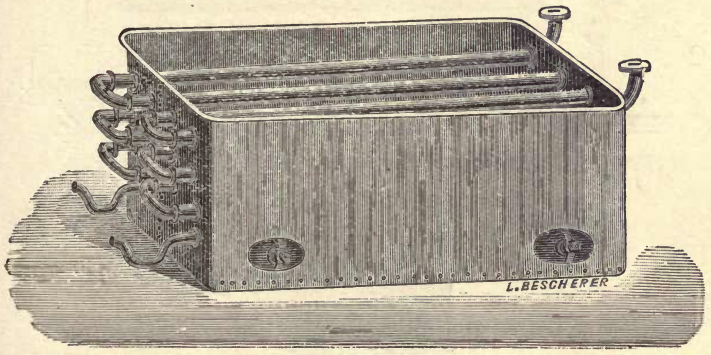

Fig. 8.—Horizontal condenser for cooling distillate.

moment no other fuel was necessary. In more modern factories this system of condensation is replaced by a condenser, consisting of a rectangular wrought-iron tank (Figs. 8 and 9), containing a series of red copper tubes, slightly sloped to allow the condensed liquid to flow out. These pipes are fixed in the tank by collars bolted on the opposite short sides. They are connected up in pairs on the outside by brazed copper jackets and elbows. They are easily dismantled for cleaning. A current of cold water enters by the bottom and issues through an overflow pipe situated a few centimetres below the upper edge of the condenser. Meyer's tubular bundle condensers are either vertical or horizontal. With a maximum condensation they consume but little water. As shown in Figs. 10 and 11, they can easily be cleaned by undoing the door at one end. As they issue from the condenser the liquid and incondensable gases pass to a separator and gas washer, thence they pass into the furnaces, as will be seen farther on. Fig. 12

shows a wooden condenser installed at the end of the large stack furnaces taking 300 stères of wood, in which the gases are propelled by a fan. So as to diminish the loss due to entrainment by the permanent

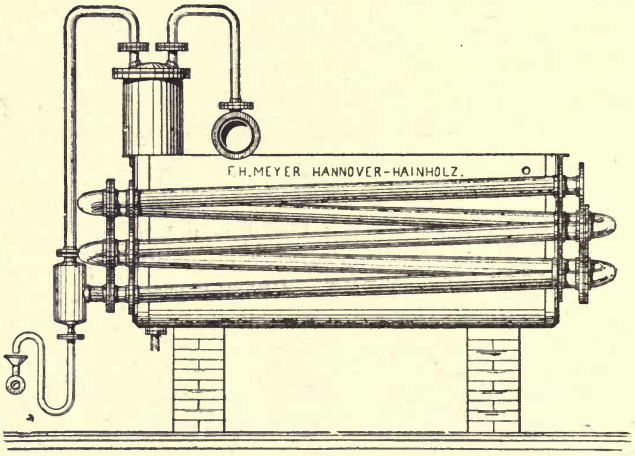

FIG. 9.—Condenser (cooler) with its separator and gas collector.

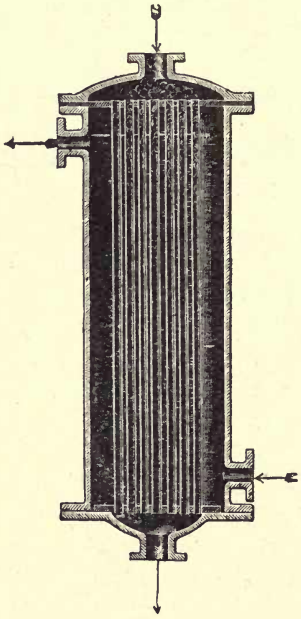

FIG. 10.—Tubular condenser (cooler), vertical.

gases, which always remain saturated with vapour, Vincent, by a special arrangement, which gives excellent results, utilised the cold, produced

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by dissolving certain salts used in the manufacture of the derivatives, carbonate of soda for instance. He passes the cold gases, as they come from the condenser, through a layer of carbonate of soda 70-80 cm. deep, spread on a double bottom in a copper column. Under the influence of the current of gas saturated with water and with acid, the crystals of carbonate of soda gradually melt and become converted into acetate

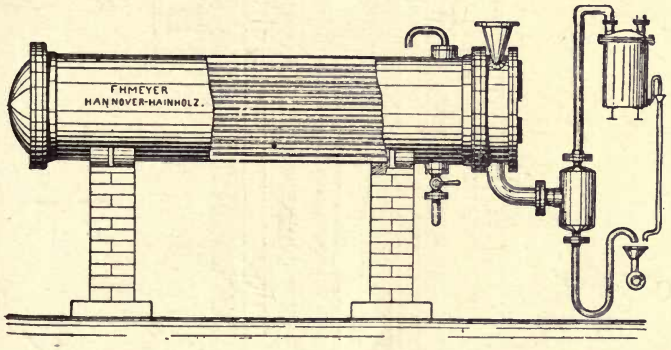

FIG. 11.—Tubular condenser (cooler), horizontal.

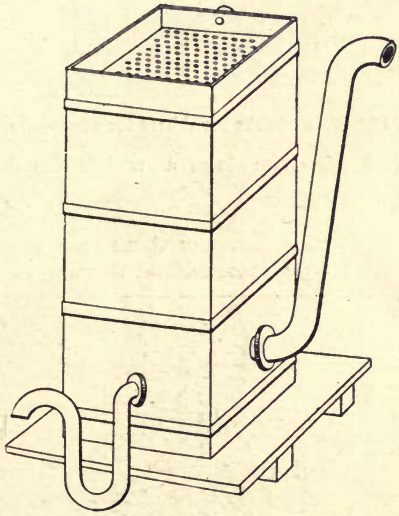

FIG. 12.—Wooden condenser for large stack ovens.

of soda, producing at the same time a considerable lowering of temperature, which causes an abundant condensation of utilisable vapours. The liquid, drawn off by a tap placed in the lower part of this apparatus, is afterwards distilled and yields crude methyl alcohol, which is condensed, and an alkaline residue, which is used in making acetate

of soda. In more recent installations they simply pass the gases into one or two coke towers (Fig. 13) through which water or weak pyroligneous acid trickles from above. As they issue from these towers the

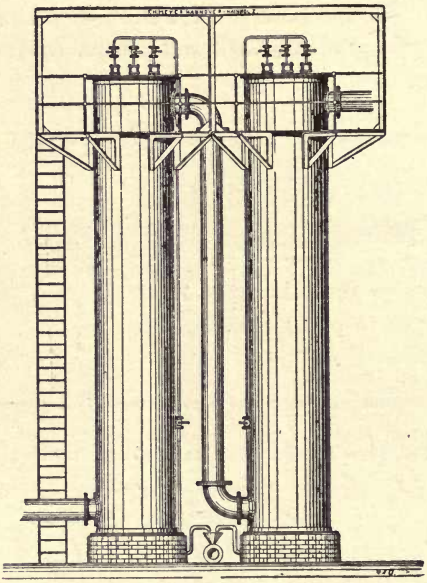

Fig. 13.—Pair of coke scrubbers for condensing gas.

gas contains, according to the stage at which the distillation of wood has arrived, the following :—

Table VII.—Showing the Composition of Gases from Wood Distillation after Passing through Coke Scrubbers

	In Beginning, per cent.	At End, per cent.
Carbonic acid, CO_2	44·90	29·20
Carbonic oxide, CO	36·80	34·90
Hydrogen as hydrocarbides	16·80	34·20
Nitrogen	1·50	1·70
	100·00	100·00

They are then made to pass by means of a Koerting injector into a lime aspirator which retains carbonic acid, whence they pass to the furnaces to be burnt.

Installation of a Wood Charcoal Factory. General Considerations.—Many things have to be considered in choosing a site for a wood carbonisation factory. Besides raw material, such as wood, coal and

lime, which enter into this industry, the facilities for transport both by rail and water which would be at disposal must be taken into account as well as the water supply, the requirements for which are great, especially for the condensation of the products disengaged by the distillation of the wood. Since an ideal factory is difficult to

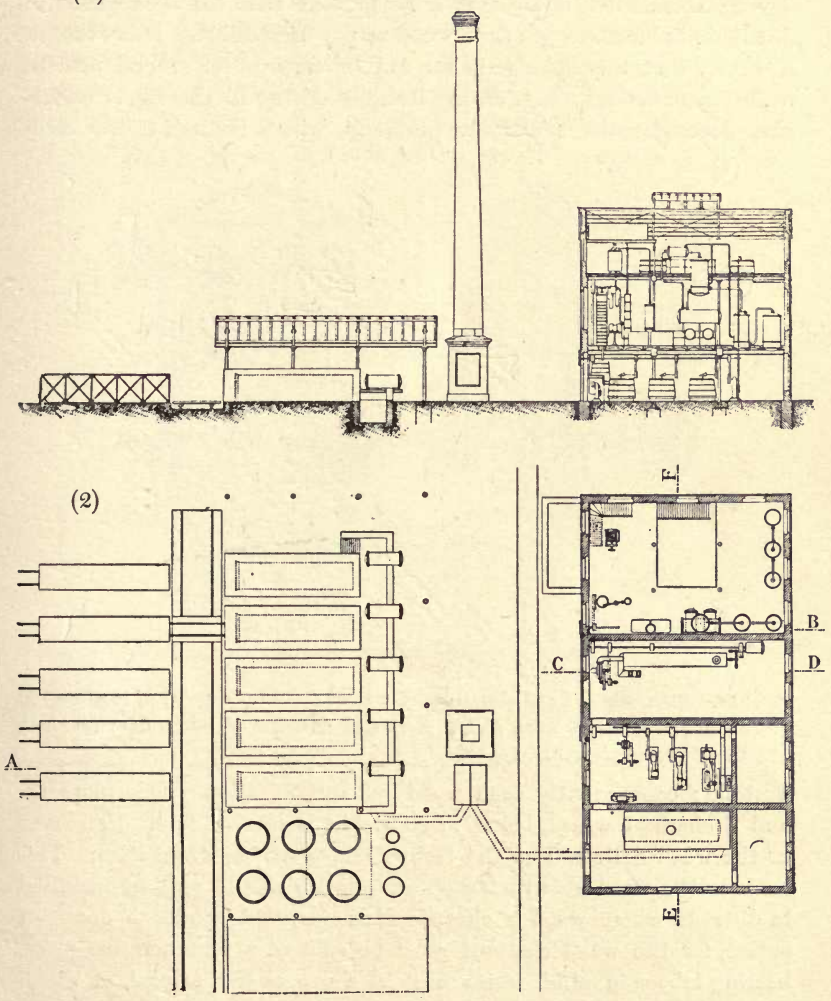

FIG. 14.—Plan, sections and elevations of a wood distillery capable of treating 100 stères (27 cords) of wood daily. (1) Sectional elevation along AB of ground plan. (2) Ground plan.

realise; it is necessary to try to get as near as possible to the forest, the wood of which it is proposed to distil, so as to reduce freight

charges of the raw material to a minimum, whilst getting at the same time near to a railway, a stream or a canal, by which lime and coal can be received and finished products despatched. A factory (Fig. 14) situated far from a wooded district would be placed at a great disadvantage, although the floating of timber could be utilised. The floating of timber as a method of transporting wood should at the present day be abandoned, because it is recognised that the best yields in distilled products are got from wood as dry as possible. It is evident, however, that this floated timber can be stacked for several months before manufacture; but, during its slow drying in the air, it undergoes decomposition, sometimes profound, which renders it less com-

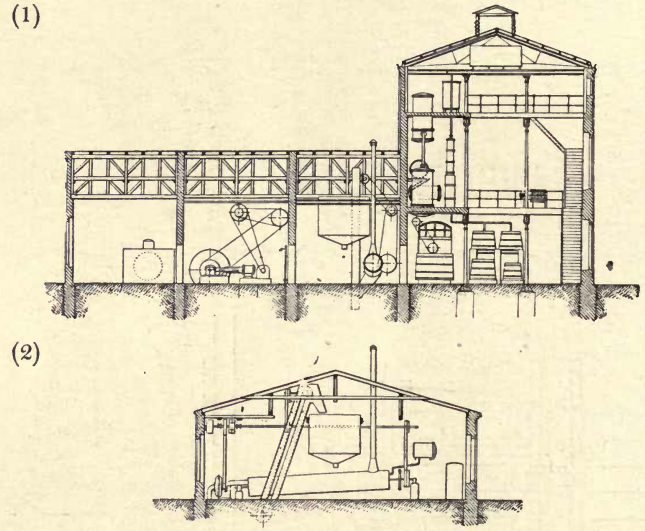

FIG. 14A.—Plan, sections and elevations of a wood distillery capable of treating 100 stères (27 cords) of wood daily. Sectional elevation (1) along CD; (2) along EF of ground plan (Fig. 14).

bustible; consequently such wood on carbonisation yields more tar and a charcoal which, being more friable, is less valuable. The cost of the wood delivered at the factory is a most important item. This leads to the conclusion that such an industry should only be installed in districts where wood is cheap. However, that condition does not suffice, for the wood charcoal must be sold at a remunerative price, bearing labour in mind, which must be cheap, as well as coal, of which a great deal is consumed. It will, therefore, be necessary not to be too far removed from the chief outlets for wood charcoal, that is to say, to be in the neighbourhood of large towns, where fuel is rather dear, and not to be too far removed from industrial centres, which

consume a large quantity. Besides, such a factory should be a certain distance away from dwellings on account of the fumes given off. Likewise, as the residual waters contain a certain amount of empyreumatic products, they cannot be let flow direct into running streams until after passing through a filtration pit, which retains the oil and tars, which are collected to be burnt.

Arrangement of the Different Buildings in a Wood Carbonisation Factory.—The steam boilers and their feed tanks ought to occupy a building in the central part of the factory, around which the other buildings should be grouped. It is necessary to place the engine room (steam engine, dynamo, water pumps) as near as possible to the boilers so as to shorten the steam pipes, which are a loss of heat for

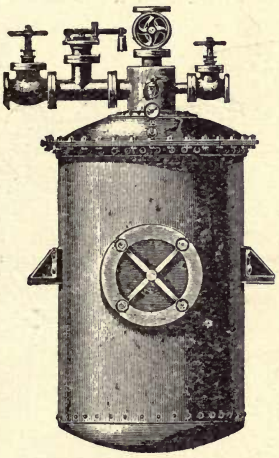

FIG. 15.—Monte-jus.

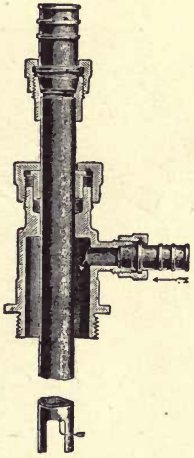

FIG. 16.—Arrangement of the plunger pipe of monte-jus.

the machines using it. In another building, still near to the boilers and not far from the buildings where the distilled products are treated—buildings which require a deal of steam—are installed the monte-jus, the pumps for crude pyroligneous products, for acids and for filter presses (Figs. 15, 16, 17). For the type of boilers, steam engines, feed pumps, etc., to select, the reader is referred to the chapter on tannic acid extracts. The retorts are placed in front of one of the faces of the quadrilateral occupied by the factory; the part of the ground where the sheds will be installed, under which the wood is stacked during the season of inclement weather whilst waiting to be distilled, will occupy a face of the factory perpendicular to that of the retorts. The wood-charcoal warehouse, as well as the space reserved for coal, is in front of the retorts. Ordinary railways for the

reception of raw material and the despatch of different manufactured products traverse these different timber yards or sheds as well as the buildings. Finally, an installation of trucks on narrow tram lines will help in the handling of all sorts of materials.

Store Tanks.—The water for feeding the boilers or for the condensers is contained in wrought-iron or reinforced concrete tanks, fixed at a certain height in the factory, for instance, above the steam engines and pumps. As the temperature and the quality of the water for the condensers plays an important rôle, it will be necessary to use well water, which must not contain any substance which can attack copper or iron. It must neither be muddy nor too calcareous, so as to avoid incrustations on the coils, which would lead to a diminished condensa-

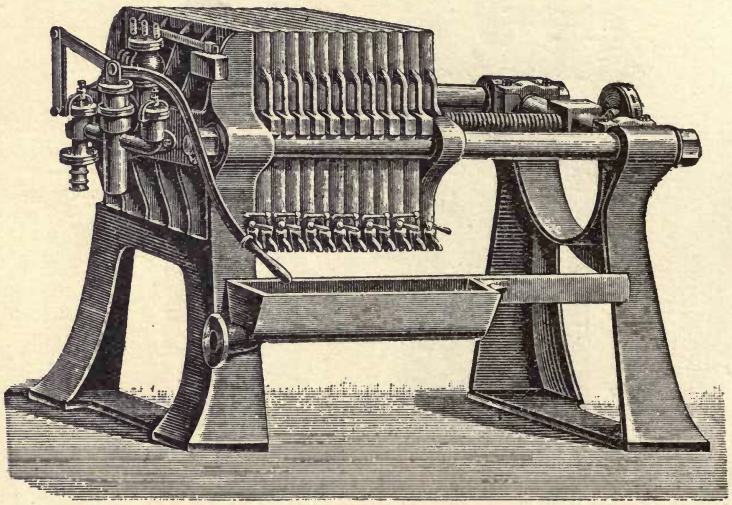

Fig. 17.—Filter press.

tion. It is calculated that a factory treating up to 100 cubic metres per day requires, on an average, 10 cubic metres of water per stère for condensation (1) of the products distilled from the carbonising retorts, (2) of the products distilled on the separation of acetic acid and methyl alcohol, (3) of the products obtained by the rectification of methyl alcohol. To feed the steam boilers, it will be preferable to possess a special reservoir which will collect the condensed steam from the different apparatus and machines where steam condenses, as in the coils of pans and stills, or from the evaporation of acetate of lime as well as the purges from the steam engine, such water having the advantage, like all distilled water, of containing no mineral matter. To the tank, which supplies the condensers with water, a draw-off pipe is

fixed for use in case of fire. Fire nozzles are installed in every building, so as to give first aid until the main pump begins to act, which may be permanently erected in the pump room connecting its force pressure with the fire pipe. It draws the water direct from the tank, or a stream, or canal. In the buildings where tars and oils are treated there ought to be a sufficient supply of sand, to extinguish in the beginning the flames of any fire that may break out. To feed the different apparatus installed in the building where the products are treated, it will be preferable to have feed tanks charged on the apparatus. These tanks, fitted with a counterpoised float, rising and falling in front of a graduated scale, are filled, when required, by pumps or monte-jus installed in the same building. Finally, tanks containing commercial products like methyl alcohol, acetone, etc., are grouped in a part of the factory far away from fire of any kind, so as to isolate them and

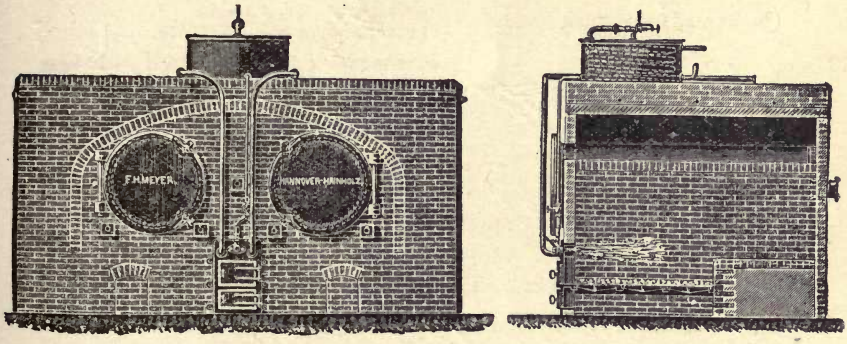

FIG. 18.—Arrangement for burning the tar under the monte-jus.

thus prevent them being set ablaze by the first outburst of a fire. Such tanks are surrounded by a small brick wall.

Use of Tar to heat Retorts or Steam Boilers.—The tar may serve, like the permanent gas, to heat the retorts and the steam boilers. Two kinds of tar have to be dealt with: (1) the tar which separates mechanically from the pyroligneous distillate; (2) the tar dissolved in the pyroligneous distillate, which is separated therefrom by the two-pan process. This latter tar, of little value, can be utilised much better as fuel, as its quality is very inferior and the amount produced is not great, from 10 to 15 kilogrammes per stère of wood. As to the first sort of tar it is generally sold, especially when it is free from water; but in certain districts, as there is no other outlet for it, it is used as fuel. The apparatus used for burning tar are analogous to those in use for petroleum residues; the tar reaches the seat of combustion by the annular space of the two concentric tubes of an injector;

the steam passing through the central tube meets the tar at the extremity of the latter, pulverises it and projects it into the furnace, bringing in its train, through the orifice fitted around the apparatus, the air required for the combustion. Two taps or valves regulate the steam and the tar. In certain installations the central tube of the injector is movable, and by means of an endless screw the flow of tar may be regulated by bringing the central tube farther forward or by shifting it farther back.

Take a factory treating 110 stères of wood weighing 380 kilogrammes, that is to say 41·8 metric tons per twenty-four hours. The products obtained resolve themselves into :—

TABLE VIII.—SHOWING RESULTS OBTAINED IN A WOOD CHARCOAL FACTORY. SOME DATA FOR ESTIMATING COST OF PRODUCTION

1. Wood charcoal : Total 12·1 tons { Large lumps, 11 tons. / Small lumps (waste), 1·1 tons.
2. Tar, 2¾ tons, which distilled gives { Water free tar, 2·2 tons. / Pyroligneous acid, 0·550 ton.
3. Pyroligneous acid, 17·600 tons, plus above 0·550 ton { decomposed in the three-pan system yields { 1·650 tons tar, 13·200 tons of a solution of acetate of lime, which yields { 2·640 tons of grey acetate of lime 80 per cent. / 5·500 tons of wood-spirit phlegms, which when rectified yields { 0·66 ton of 80 per cent. crude wood-spirit.
4. Gas, 8·550 tons used in heating the retorts.

The plant consists of fourteen movable retorts, which, by making twenty to twenty-two charges per twenty-four hours, enables the above amount of wood to be passed through them. As it possesses a rolling bridge, the hot retort is taken out of the furnace and laid on a truck to be immediately replaced by another freshly charged and brought in proximity to the spot ; the time of charging and discharging the retort does not exceed more than eight to ten minutes on an average. Fifty workmen being employed in the factory, and labour costing 1·85 to 2·45 francs per day, brings the labour to about 2·1 francs per stère of wood. This price may vary with standing-still or break-down days. The lime used per stère comes to 0·18 franc. As to coal consumed it is necessary to estimate on 100 to 105 kilogrammes per stère, which comes to 2 to 2·5 francs, taking the price of coal as 22 to 25 francs the ton according to the district in which the factory is situated. The general expenses of such a factory amount to 3·15 to 3·70 francs per stère of wood carbonised, in which it is necessary to include the interest on the capital employed, which may amount to as much as 750,000 francs (say £30,000),

of which 400,000 francs (£16,000) are for plant, 280,000 francs (£11,200) for purchasing wood, and the remainder as working capital.

Treatment of the Products obtained.—As already seen, in the description of the products obtained in the carbonisation of wood, the liquid which flows from the condenser being at one time strongly coloured, and at other times pale, care is taken to "cut" the still at the moment it shows a pale brown colour, and a high percentage of acid as compared with the liquid which previously issued from the condenser, the same precaution being taken towards the end of the distillation, when the liquid again turns brown owing to the presence of a greater amount of tar. These products, besides their colour and percentage of acidity, which the workman can determine as often as necessary by means of a solution of normal soda, are easily recognisable by the smell. In the first stage the distillate, though almost free from acid and from tar, has a feeble empyreumatic odour due to the first disengagement of gas; in the second stage the distillate is dark, without, however, containing, for all that, a large proportion of tar, and the quantity of gas produced is greater, but diminishes gradually thereafter, whilst as the third stage is entered the proportion of tar increases. Finally, in the last stage only a small amount of gas is disengaged, the distilled products flow drop by drop from the condenser, and the goose-neck cools. By means of a series of gutters or pipes the condensed products, according to their nature, are run into different reservoirs, the upper portion of which is below the mouth of the condenser exit pipe, so as to let the liquids flow into them naturally. These products, left to settle in the vats for some time, separate into three distinct layers, the lower layer consisting of tar charged with creosote oil and with acetic acid, the middle layer containing water, pyroligneous acid, wood-spirit, acetone and some tarry matter held in solution by the aid of the acetic acid and wood-spirit, finally the upper layer, consisting of light hydrocarbides, still holding acetic acid in solution. The aqueous liquid is separated mechanically from the tars and from the heavy oils through taps placed at different levels; each layer is filtered through a bed of coarse sand, from which they flow into different receptacles, whence they are taken up by pumps and pumped into special reservoirs specially retained for each of these three products.

Treatment of the Pyroligneous Distillate.—The pyroligneous distillate, thus freed from the bulk of the oils, is afterwards run into stills to be separated into pyroligneous acid (or one of its compounds), wood-spirit and tar. To effect this separation several methods are adopted. In some the liquid simply undergoes fractional distillation, in others the pyroligneous acid is neutralised by an alkali or an alkaline earth before

distilling it to separate the wood-spirit. Finally in a third style of working the whole is distilled—water, wood-spirit and pyroligneous acid; the condensed products are then collected over lime, then they are redistilled to separate the alcohol.

(1) *Simple Distillation.*—The vessels used to separate pyroligneous acid from wood-spirit are always copper ones; generally a still is used of 3 metres capacity, heated by naked fire or by steam. The first portion of the distillate, containing wood-spirit, acetone, but little acetic acid, traverses three copper rectifying plates cooled on the outside by a small but calculated quantity of water. In this way a liquid rather rich in alcohol is obtained, which is collected in the crude alcohol tank so that it may afterwards undergo another treatment, which will be studied later on. If it be merely desired to make pyrolignite of lime or pyrolignite of soda, the distillation is stopped when all the wood-spirit has passed over, then after cooling for some time the product is

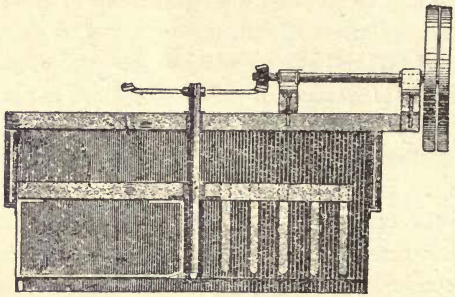

Fig. 19.—Mixing vat for neutralising the pyroligneous acid fitted with mechanical agitator.

run off through a tap in the bottom; a mixture of water, acid and tar runs off first, and is collected apart, then the pyroligneous acid, which is run into tanks, where it is saturated by carbonate of lime or by carbonate of soda (Fig. 19). But if it be desired to produce pyroligneous acid to make pyrolignite (sugar) of lead, for example, the distillation is continued, taking care to separate the acid liquid, which condenses, and to run it into another reservoir. In all cases when oily patches float on the condensed liquid the distillation is stopped, for nothing then remains in the still except tar, which is collected along with the other tar, to be treated later on, for the extraction of creosote, for instance.

(2) *Distillation of the previously neutralised Pyroligneous Distillate.*—In this process, employed generally in the direct manufacture of pyrolignite of lime, a predetermined volume of milk of lime, calculated on the amount of acid to be neutralised, is run into a still fitted with an agitator—the acid contained in the pyroligneous distillate

having been titrated beforehand. After the mass has been well mixed and it has been made sure that the liquid is neutral to litmus paper, the still is heated, preferably by steam, whilst the agitator is still kept at work. A mixture of water, methyl alcohol and acetone distils; the liquid issuing from the condenser is collected, according to the stage the distillation has reached, in one of the two tanks reserved for phlegms, so as to separate the first running's phlegms from the last running's phlegms as soon as the condensed liquid turns milky. The distillation is finished when a sample of the distillate, examined by

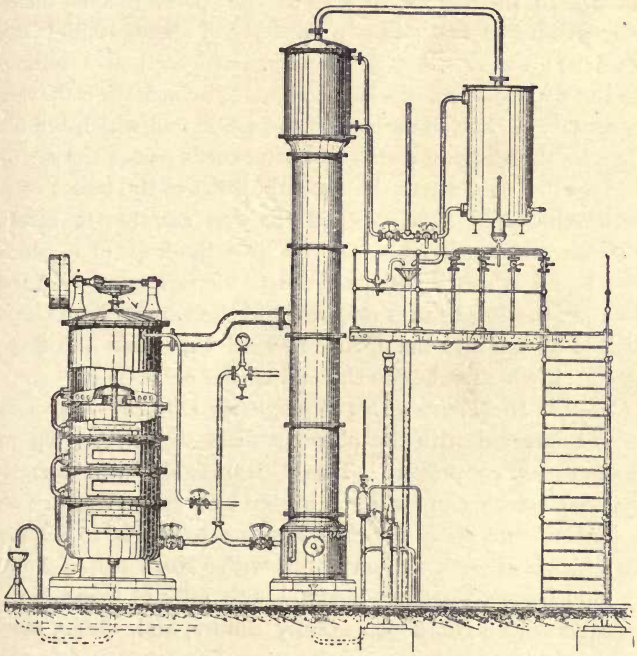

FIG. 20.—Set of stills with mechanical agitators adapted for continuous working and connected with rectifying column for the distillation of the previously neutralised pyroligneous distillate.

the alcoholometer, shows no more alcohol, or when a few drops of the sample, thrown on red-hot coal, appear to be water free from alcohol. The residue in the still is then run into the evaporation pans, where it is concentrated preferably by the waste heat of the furnaces. When it is desired to convert pyroligneous acid into pyrolignite of soda, it is often separated from the wood-spirit by distilling the crude liquid—freed from tar—in two consecutive pans connected with each other; the second pan, fitted with an agitator, contains certain amounts of lime and of sulphate of soda dissolved in water, the amounts being

determined after titrating the pyroligneous acid introduced into the first pan. The acid products distilled from this pan by a steam coil are converted in the second pan into acetate of soda, owing to the affinity of the sulphuric acid of the sulphate of soda for the lime, whilst the alcohol, acetone and their non-acid derivatives are again distilled and condensed. The distillation finished, the mixture from the second still is run into a wrought-iron reservoir, where it deposits the sulphate of lime produced in the process; the clear liquid is then run off and evaporated. In the three-pan system the pyroligneous distillate is heated by steam in a first still of a capacity of about 10,000 litres (say 2,200 gallons); the distilled products are collected in a milk of lime contained in a second pan, in which the methylic acetate is decomposed, forming acetate of lime, likewise also the acetic acid which bubbles into it. The wood-spirit again distils and afterwards passes through a milk of lime in a third pan, where the decomposition of the traces of methyl acetate which might have escaped the first reaction is terminated. Finally steam and methyl alcohol run into the coil of a condenser; when the liquid which flows from the condenser marks zero on the alcoholometer the third pan is stopped and the vapour from the second is allowed to escape into the open air, until that from the first pan is no longer acid, which indicates the end of the operation.

(3) *Complete Distillation of the Pyroligneous Distillate.*—By a final arrangement connected with the above process the distillation may be made, so to speak, continuous. Two stills of 6,000 litres capacity (say 1,320 gallons), working in turns and heated by a steam coil, are charged from a higher level with the pyroligneous products to be distilled. The still which is at work communicates with a No. 1 pan of 2,500 litres capacity (550 Imperial gallons) charged with milk of lime; No. 1 pan communicates with a No. 2 pan exactly similar, and which, moreover, is in communication with the condenser. The vapours (steam, pyroligneous acid, wood-spirit, acetone, etc.) which come from the still charged with pyroligneous products, are led by a pipe pierced with holes to the bottom of No. 1, which retains the bulk of the acid products; as No. 1 is also heated, a fresh distillation takes place, and the distilled products, still containing pyroligneous acid, now bubble into the milk of lime in No. 2, which should only allow acid-free substances, like wood-spirit and acetone, to pass through it. These are liquefied in the condenser, from which they pass to a special reservoir, the liquid collected in which then contains 30 to 40 per cent. of alcohol. From time to time the contents of No. 1 pan are controlled by taking a sample from the test tap, and as soon as the test shows a slight acidity, No. 1 pan is isolated by manipulating certain taps so as to put the still

in direct communication with No. 2. Then No. 1 is emptied of its contents, which are run into a vat. No. 1 is then charged with fresh milk of lime to work in its turn, by a play of taps, as the No. 2 pan, for the absorption of acid vapours. The vapours, free from acid, then pass to the condenser or into a distilling column, in which the methyl alcohol is concentrated (Fig. 21). When there is no more acid and consequently no more alcohol in the first still, a fact which is ascertained by testing, it is disconnected from the milk of lime pan, which is then connected with the second still, and this is at once started to work. For the transfer of the crude pyroligneous distillate, pumps or

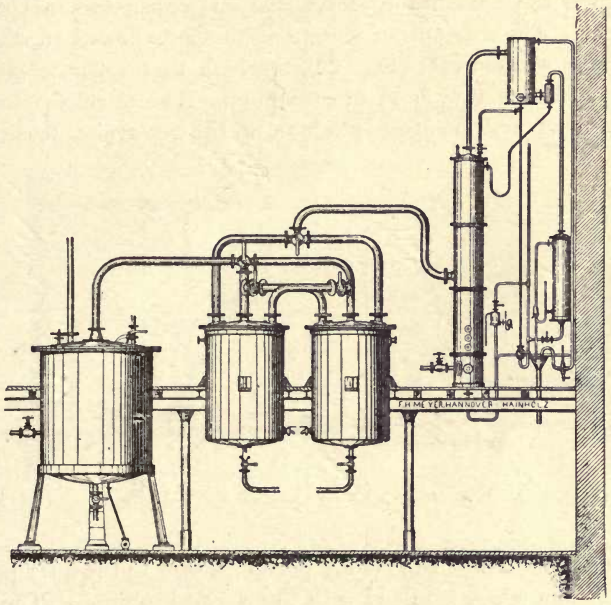

FIG. 21.—Group of three stills for continuous working with rectification plant for the complete distillation of the pyroligneous distillate.

monte-jus are preferable to injectors, which have the defect of being liable to become obstructed by the tar which remains suspended in the liquid and might perhaps occasion serious accidents to the distilling plant. In fact, the pipe which brings the pyroligneous distlilate from the injector being obstructed, steam alone continues to pass into the pan until the moment when the pipe begins to clear itself, the liquid suddenly runs into the pan, cools the latter, a comparative vacuum is formed, and the pan is flattened.

Pyrolignite of Lime. Treatment of the Acetate of Lime Solution.—With methyl alcohol and charcoal, pyrolignite of lime is one of the

most important products of a wood charcoal factory. Great care, therefore, should be exercised in its manufacture, not only to reduce loss in manufacture as far as possible, but also to obtain a product rich enough in acetic acid (60 and even 65 per cent.), and containing at the same time a minimum of free lime and tarry matter, the latter giving rise to some difficulties in the manufacture of commercial acetic acid. That explains the reason why the manufacturer is obliged to separate the condensed products obtained in wood charcoal manufacture into respectively: (*a*) pyroligneous products for grey acetate of lime manufacture, and (*b*) pyroligneous products for brown acetate of lime manufacture, the amount of acetic acid in which does not exceed 4·5 per cent. The solutions of acetate of lime are allowed to stand for some days in wooden vats (Fig. 22), in which they separate from the tar and mud which they hold in suspension. The clarified solution is then drawn off and pumped into one of the reservoirs, feeding the

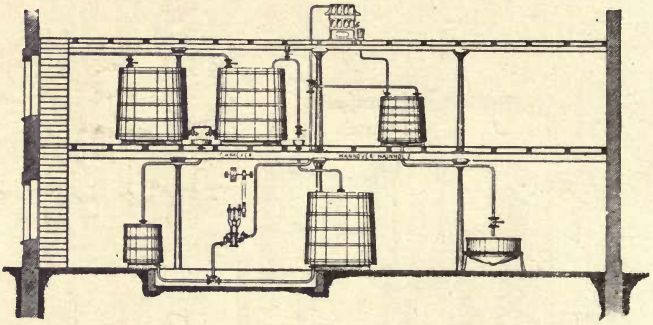

Fig. 22.—Installation of clarifying and evaporating vats and filter press for acetate of lime manufacture.

evaporating stills, whilst the muddy portion is sent to the filter presses, the filtrate from which is mixed with the clarified solution. The wash water, from washing the cakes in the filter presses, is utilised for making milk of lime. The 20 per cent. acetate of lime solutions cannot be evaporated and set aside to crystallise like an ordinary mineral salt; therefore different systems of concentration are used to convert it into a commercial, and more especially a transportable, product. In some factories it is evaporated in hemispherical cast-iron pans heated over a naked fire; during the whole of this operation, in consequence of the action of the air on certain organic matters, tarry scums form on the surface of the liquid, which have to be skimmed off, in order to facilitate evaporation. When the solution reaches 15° B. (23° Tw.), sp. gr. 1·115, crusts of acetate of lime become attached to the sides of the pan. This crust must be removed to prevent the decomposition

of the salt by overheating. Finally, when the whole mass is sufficiently evaporated and has a pasty appearance, the fire is withdrawn from below the pan, then the drying is finished, by small quantities at a time, in two flat cast-iron pans continuously heated by the same furnace. In these pans the salt is constantly stirred by an iron spatula to avoid all decomposition. The acetate of lime undergoes preliminary drying in the pan farthest from the fire, then it is placed in that directly over the fire, where the drying is finished, whilst the first pan is refilled with paste in order to start a fresh charge. Certain manufacturers prefer to lift the crystalline crusts, which form in the evaporation of acetate of lime solutions, by a shovel. These crusts are laid to drain in a basket placed above the pan, to be afterwards dried and piled on a cast-iron sole-plate, heated by the waste heat of the distillation furnaces. In this process, pending the final drying, which takes place in the hottest part of the stove, care is taken to stir the acetate continu-

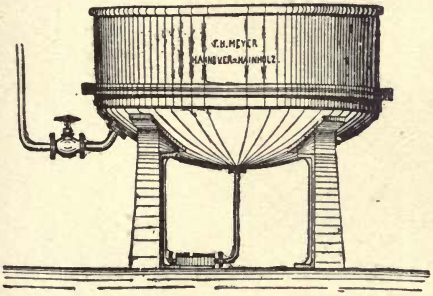

FIG. 23.—Steam-jacketed pan for evaporating solutions of acetate of lime.

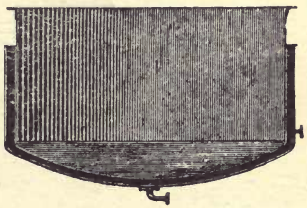

FIG. 24.—Steam-jacketed pan for evaporating solutions of acetate of lime, section.

ally by means of special appliances, so as to crush the salt and to prevent it from agglomerating on the side of the pan. In another process the pans are arranged in ascending order directly over a fire; the one farthest away is used to concentrate the acetate of lime, whilst the pans nearer the fire dry the salt completely. This plan, which needs much room and does not sufficiently prevent the decomposition of the acetate of lime, is often modified by the use of steam-heated, jacketed, copper or wrought-iron flat pans of 2,000 litres (440 gallons) capacity (Fig. 23). Steam coils do not suit this class of evaporating pan, for they render the stirring of the mass difficult, especially when the pellicles of acetate of lime begin to form. Round, wrought-iron or rectangular pans with cast-iron bottoms and coils (Figs. 24, 25, 26), issuing from the cast-iron bottom, are likewise successfully employed.

The evaporation of the solution of acetate of lime in this kind of pan goes on comparatively quickly up to 40 per cent. using ordinary

steam, but it is then necessary to stir the mass with mechanical agitators; for that purpose small pans requiring less power, because the mass to be set in motion has less bulk, will be chosen in preference to large pans, which require a greater expenditure of steam, not only to drive the mixer but to finish drying. It is better not to complete the desiccation in these pans. When the acetate of lime turns brown and becomes pasty between the fingers without adhering, it is dried completely by projecting it on to cast-iron plates heated to redness by the gases from the furnaces of the wood charcoal retorts. Acetate of lime may also be drained in the concentrated pasty condition in stoves, heated by a current of hot air, the circulation of which is facilitated by

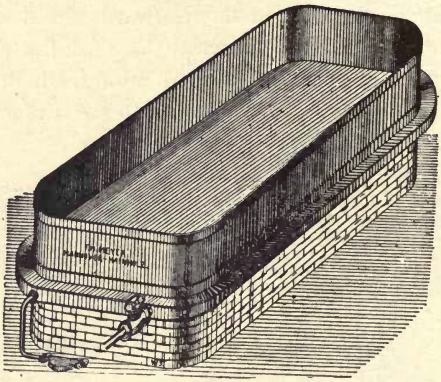

FIG. 25.—Shallow pan heated by coil for evaporating taceate of line.

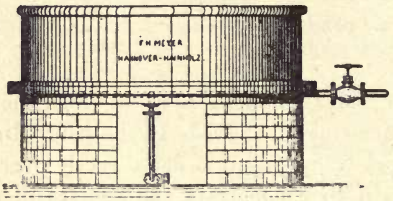

FIG. 26.—Shallow pan heated by coil for evaporating acetate of lime.

fans, or the acetate of lime may be spread in thin layers on wrought-iron plates heated by a special furnace, the gases from which circulate in winding flues. By this process the still moist acetate of lime, being placed above the furnace itself to avoid overheating, winds round with the flues so as to give to these torrefiers the form of a gridiron. The torrefaction is intended in the first place to eliminate the last trace of water, so as to get an 80 per cent. acetate; in the second place it removes the volatile tar oils, which, unless this were done, would impart a dark brown colour and empyreumatic odour to the acetate of lime. As soon as the acetate of lime has assumed its grey

colour and is easily reduced to powder between the teeth, torrefaction is complete; the product is then ground to small lumps by means of a hand roller which crushes it. The installation of a mechanical drier (Fig. 27) in the form of a drum, requiring so to speak but little labour, has the advantage of yielding a more uniform product without waste, due to the carrying away by the air of dust formed during drying. The acetate of lime to be torrefied is led into the drum by the extremity opposite to that by which the hot air enters, and is put in motion by a helix, which moves in a cylinder surrounded by hot air. As it issues the acetate of lime goes up the cups to a floor above, into a sort of wooden bin, from which it is bagged up into sacks of 50 and 70 kilogrammes (110 and 154 lb.). This acetate of lime resembles the grey acetate of lime of commerce; it contains about 10 per cent. of water, a maximum of 84 per cent. of pure acetate of lime and 6 per

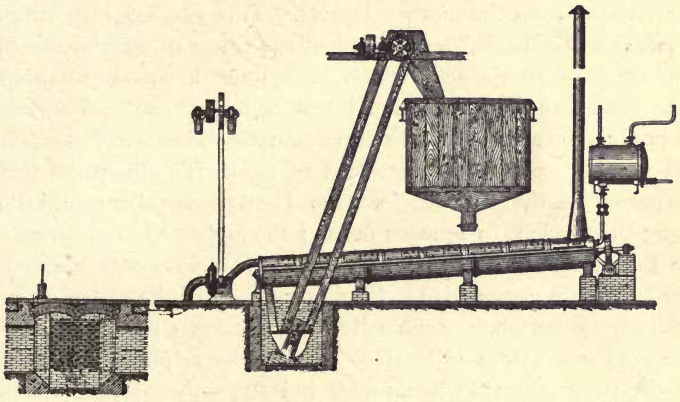

FIG. 27.—Drum drier for acetate of lime.

cent. of foreign bodies: tar, lime, carbonate of lime, etc. Of the 10 per cent., 4 to 6 per cent. may be eliminated at 150° C. (302° F.), and the remainder at a higher temperature, but there is no advantage in exceeding an acetate content of 80 to 82 per cent., because it would be at the expense of the yield; this product commencing to decompose at 150° C. (302° F.).

Brown Acetate of Lime.—This salt is obtained by saturating the dark brown pyroligneous distillate from wood by lime. As the liquid contains dissolved tar, it follows that the brown acetate so obtained has a much lower acetate of lime content (only 45 per cent.) than the grey acetate. This acetate, being less easily decomposed by mineral acids than the grey acetate, it follows that the value of the acetic acid made from it is less than that of the acetic acid made from grey acetate.

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CHAPTER III

THE ACETIC ACID INDUSTRY

Acetic Acid from Acetate of Soda.—Formerly acetic acid was made by wood distillers; they first converted the acetate of lime into acetate of soda, as previously indicated, or prepared acetate of soda directly from the pyroligneous distillate and carbonate of soda. The soda solution was concentrated so as to obtain crystallised acetate of soda, which was separated from the mother liquors. This purified salt, known in commerce as "red salt," on account of the traces of tar and the higher homologues of acetic acid which it contained, was decomposed by sulphuric acid and yielded a liquid testing 50 per cent. of acetic acid. This product, on account of the empyreumatic bodies which it contained, could not be used as an ingredient of food. To obtain the acid in a more pure state, the "red salt" was fused in its water of crystallisation, by heating the product to igneous fusion; the butyrates and propionates were thus decomposed; then the acetate of soda was taken up by water and again crystallised. By a new fusion of the salt, followed by a fresh crystallisation, a chemically pure salt was obtained, from which acetic acid was extracted by decomposition by sulphuric acid. It was only the progress made in another industry—the alcohol industry—which led manufacturers to produce different qualities of acetic acid capable of being used in certain industries. At the present day commercial acetic acid, termed acetic acid for industrial purposes (*acide acétique des arts*), the greatest outlets for which are verdigris, chrome yellow, white lead, aniline, etc., is prepared from acetate of lime, the presence of certain organic matters which accompany it having no very bad effect.

Acetic Acid from Acetate of Lime.—Two mineral acids are used to decompose the acetate of lime: (1) commercial hydrochloric acid; (2) commercial sulphuric acid of 60° to 66° B. (142° to 168° Tw.), sp. gr. 1·715 to 1·84. The two processes are both used, and the selection of either depends on the quality of acid desired and the circumstances in which the manufacturer is placed: purchase of raw materials, outlet for acetic acid produced, etc. In fact, according to the products which it is desired to manufacture, the following strengths and grades are

required: (1) commercial acetic acid of 38, 60 or 75 per cent.; (2) concentrated commercial acetic acid of 85 to 100 per cent.; (3) fine tasted acetic acid (*acide acétique bon goût*) for dietetic purposes, free from metallic compounds and empyreumatic products; (4) chemically pure acetic acid of 96 to 100 per cent. For the production of industrial acid, the commercial hydrochloric acid process is preferred, being much more economical than the sulphuric acid process, which always requires mechanical agitators.

(1) *Hydrochloric Acid Process.*—The manufacture of acetic acid by hydrochloric acid may be carried out in two ways: the first, which is the oldest and still the most used, was discovered almost simultaneously by Winkel and Christi. It consists in mixing hydrochloric acid with the acetate of lime, and allowing the products of the reaction to settle for twelve hours. Wooden vats or pits built of masonry and fitted with an agitator may be used. For 100 kilogrammes (220 lb.) of acetate of lime, there is used, according to its acetic acid content, from 95 to 115 litres (20·9 to 25·3 gallons) of hydrochloric acid of 20° to 21° B. (32° to 34° Tw.), sp. gr. 1·16 to 1·17. The acetate of lime being in the vat, the acid is run on to it and the whole stirred until it is completely fluid; it is then left to stand for several hours. A certain amount of tarry matter separates, which rises to the surface of the liquid and is removed by a large skimmer. The solution of chloride of calcium formed in the acetic acid, of a density of 1·250, is then run into a still, consisting of a copper pan heated by naked fire or by an open steam pipe and a closed steam coil, which are fitted up in its interior. This apparatus is in communication, by a rather wide pipe, with a copper or, better still, a tin condenser.

If heating be done by steam, distillation is commenced by passing waste steam from the pumps or the engines through the coil; about 50 per cent. of the product is distilled in that way; then direct steam is turned on through the "blow-up" pipe; the steam carries over the rest of the acetic acid, and leaves in the still an aqueous solution of chloride of calcium and tarry matter, rendered insoluble in consequence of the departure of the acetic acid. This residue is then run out through a pipe situated in the lower part of the apparatus. The acetic acid obtained by this process titrates from 40 to 45 per cent. and may be used for many purposes; but when intended for certain industries which require it free from chlorine, it has to be redistilled with lime (the amount of which has been determined from the titration of the chlorine as silver chloride). This operation is conducted in a pan similar to the foregoing, but capable of being heated by a steam jacket. As the products which distil in the beginning and

at the end of the operation are milky, from the empyreumatic oils which they hold in suspension, they are separated from the middle runnings and allowed to stand; after running off the oils, which float to the top, the acid is mixed with the products to be rectified in a fresh operation. By the distillation of 100 kilogrammes of 82 per cent. acetate of lime, 60 kilogrammes of acetic acid of 100 per cent. strength should be obtained, but this result is never realised, because the solution of chloride of calcium always retains a little acetic acid, the cost of extraction of which would exceed its value. When 85 to 100 per cent. acid is desired, a copper rectifying column (Fig. 28) consisting of a cylindrical pan of about 8 mm. (say $\frac{1}{3}$ of an inch) thick, with an average capacity of 3,000 litres (660 gallons) and heated by a steam

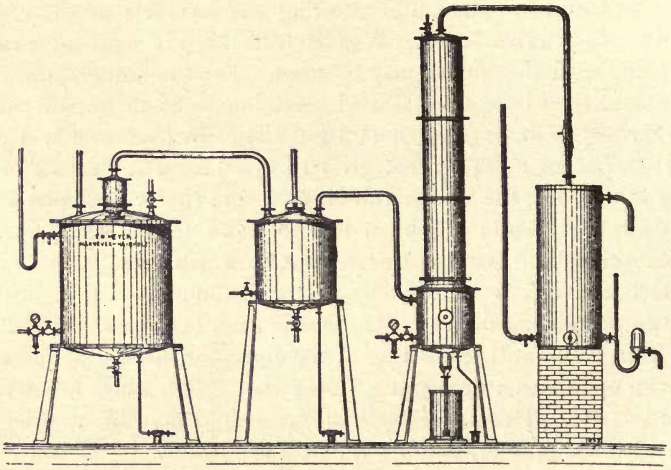

FIG. 28.—Plant for the continuous manufacture of commercial acetic acid of 85 to 100 per cent. strength.

coil is used. The column is similar to that of an ordinary alcohol rectifier, in which copper plates are replaced by porcelain or earthenware plates. The strength of the acid as it issues from this column is higher the weaker the current of water which circulates on the plates, and, as it is generally desired to obtain the crystallisable acid, not because it is more valuable but because it is more pure (the presence of the vapour of water in the distillation aids in the carrying away of propionic and butyric acids), the flow of water on the plates should be regulated so as to collect, after some time, crystallisable acid of 96 per cent. strength: 100 lb. of 45 per cent. acid yield on rectification 25 to 30 per cent. of 96 per cent. acid, and 75 to 80 per cent. of 20 per cent. acid. The distillation of acetic acid in solution with chloride of calcium may be carried out continuously according to Boessneck's

process. The solution, previously warmed in a copper pan, flows in a continuous manner on to the top part of a column apparatus. As it falls from plate to plate, the acetic acid separates and passes into a condenser, where it is liquefied, whilst a solution of calcium chloride flows away from the bottom of the column. The strength of the acid obtained in this way is 45 to 50 per cent. and is of sufficient purity.

(2) *Sulphuric Acid Process.*—In this process the plant employed is altogether different from the preceding, because mechanical mixers, which were not required for the distillation of the mixture of acetate of lime and hydrochloric acid, which always remains fluid, become indispensable by the use of sulphuric acid which yields an almost solid mass with acetate of lime. Another peculiarity of this process is that sulphuric acid, acting in an aqueous medium, gives rise to secondary reactions; in fact, owing to the heat disengaged, but still more to the heat radiated from the masonry surrounding the pan, since it is heated by naked fire, sulphurous acid (arising from the action of the sulphuric acid on the tars) is generated; moreover, the oils present in the pyrolignite of lime distil with the acetic acid. The crude acetic acid obtained by the use of sulphuric acid is, therefore, more impure than that made by the aid of hydrochloric acid, but it has the benefit of being more concentrated, and that is readily understood, because if it be taken that 100 lb. of pyrolignite of lime still contains 10 per cent. of water for 86 per cent. of pure acetate of lime, and that the 60 lb. of sulphuric acid, theoretically necessary, brings 7 per cent. of water, about 70 lb. of 85 per cent. acid should be collected, but this result is never obtained, especially in using distillation with access of air by direct heat, and still less if only the theoretical amount of sulphuric acid be made to intervene. In fact, to prevent the acetate of lime from being attacked by the sulphuric acid and thus converted into acetone, an excess of 8 to 10 lb. of sulphuric acid per 100 lb. of lime should be added to replace the acid which is converted by the tar into sulphurous acid, as already mentioned. The acetic acid obtained, termed *acide acétique des arts* (commercial acetic acid), only tests about 75 per cent., because it contains not only the water brought by the sulphuric acid and the pyrolignite, but also that coming from the reduction of the sulphuric acid by the tar.

Merits and Defects of the two Processes.—Besides the difference in the acetic acid content of the products got by the two processes, that by hydrochloric acid yielding 45 to 50 per cent. instead of 75 per cent. by the second, it is to be noted that the sulphuric acid process entails a larger consumption of fuel, owing to the power required to drive the mixers, without taking into account that the proportion of sulphuric

acid required is greater than theory, and that that is not the case as far as the hydrochloric acid process is concerned. On the other hand, the weight of raw materials with which the vessels are charged must be less in the second process than in the first, hence increase in manufacturing expenses, and again, after the distillation of the acetic acid produced by the decomposition of pyrolignite by hydrochloric acid, a fresh distillation of the acetic acid may be necessary to get a product free from mineral acid. Putting the above drawbacks of the sulphuric acid process to one side, it has an advantage which is not unimportant if residuals must be taken into account; in fact, chloride of calcium is a product which, beyond its minimum use for freezing mixtures for cold-storage installations, becomes a cumbersome liquid if authority

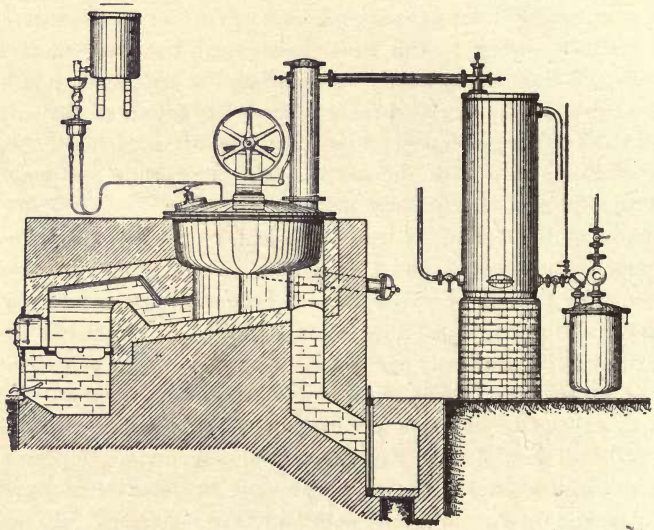

Fig. 29.—Plant for the manufacture of acetic acid by the sulphuric acid process under atmospheric pressure.

cannot be got to run it into a running stream, while sulphate of lime can be spread on arable land poor in lime.[1] It follows from these considerations that when it is a question of producing industrial acetic acid it may be preferable to use sulphuric acid. The plant used for the manufacture of acetic acid, by the decomposition of pyrolignite of lime by means of sulphuric acid, consists of a flat cast-iron pan heated by a naked fire (Fig. 29). This pan, built into the masonry of the

[1] But in London, for instance, residual sulphate of lime has to be barged out to sea and discharged beyond reach of tide, whilst possibly no objection would be taken to running the calcium chloride into the Thames. There is some possibility of an outlet for calcium chloride as a dust preventive for roads if the objections of the proprietors of river and lake fisheries do not stop its use.—D. G.

furnace, has a pipe in its bottom through which the residue may be evacuated when the operation is finished. An agitator with blades keeps the mass in motion from the commencement of charging, first with pyrolignite of lime, then with sulphuric acid, until the operation is terminated. The acetic acid distilled, after having traversed a dust chamber, passes to a condenser, where it is condensed.

Van der Linden's Process.—This process (French patent 248,056, 10th January, 1895, with certificate of addition of 14th April, 1896; German patent 92,418, class 12, deposited the 29th November, 1894) is a very ingenious modification of the manufacture of acetic acid from pyrolignite of lime or other acetate and sulphuric acid (Figs. 30, 31 and 31A).

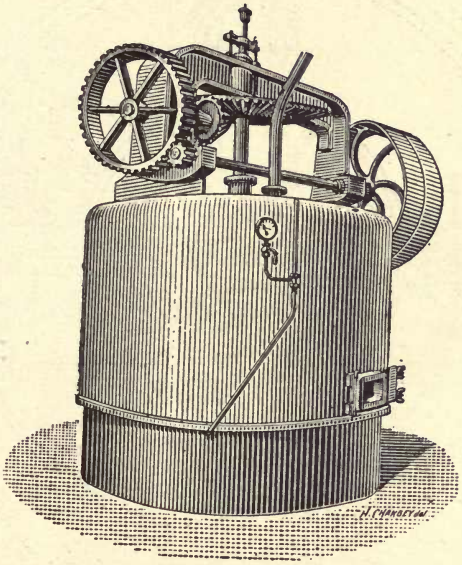

FIG. 30.—Apparatus for the manufacture of acetic acid by Van der Linden's process.

The distillation is conducted under reduced pressure, and a steam heat may be used instead of a fire underneath the pot. Under the action of the vacuum the acetic acid distils at a lower temperature, the advantage of which is that (1) secondary products due to the action of the sulphuric acid on the oils or tars in admixture with the pyroacetate of lime are avoided; (2) a less amount of sulphuric acid suffices than when distillation is conducted under the ordinary atmospheric pressure, an amount which approaches the theoretical amount of sulphuric acid required for a complete reaction. The acetic acid is therefore more pure, since it contains no empyreumatic products. Moreover, capacious vessels may be used, capable of treating rather heavy mixings, as in

the hydrochloric acid process, for example, and also of treating either brown or grey pyrolignite of lime.

Behren's Process.—Consists in mixing pyrolignite of lime with acetic acid and then treating the mixture with sulphuric acid. It is thus possible, by diluting the mass, to use only the theoretical amount of sulphuric acid required to decompose the acetate without producing secondary reactions. One part of acetate of lime of 81 per cent. strength is stirred into 1 part of acetic acid, then the theoretical quantity of sulphuric acid is added, say 0·55 part of 92 per cent. monohydrated

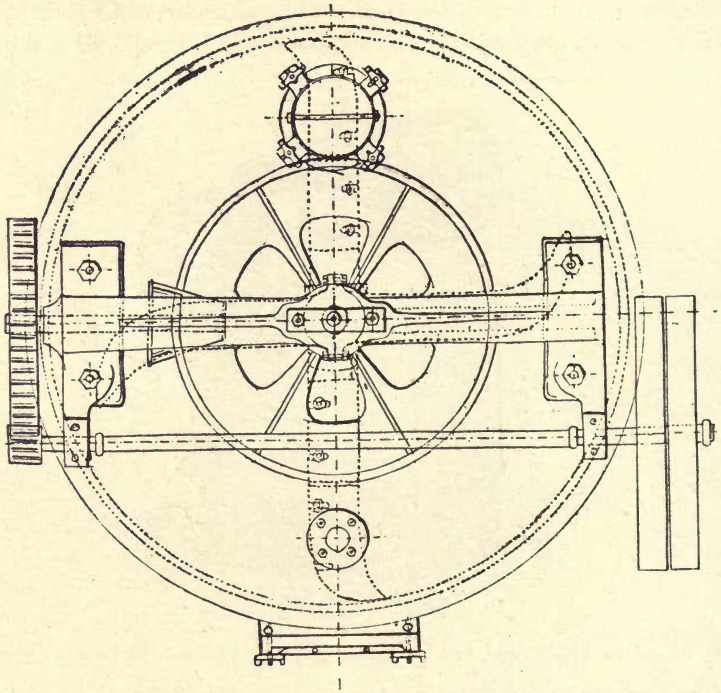

Fig. 31.—Apparatus for the manufacture of acetic acid by Van der Linden's process, cross section.

acid. The mass having been well mixed, it is passed through a filter press; the liquid which flows away is acetic acid, sufficiently concentrated to be used in the manufacture of glacial acetic acid; as to the residue it is gypsum.

Installation of an Acetic Acid Factory. Description of a Factory treating Pyrolignite of Lime by Sulphuric Acid and afterwards Rectifying the Acid Product obtained.—Such a factory will be situated at a certain distance from inhabited houses, so as not to incommode the neighbours by the strong smell given off by the residuals as they are being discharged.

The pans used for making commercial acetic acid are cast-iron ones. They are fitted with an agitator, and are heated by masonry furnaces or by steam ; in the first case, so as to ensure uniform heating, the dimensions of the pans are calculated so as to treat 500 kilogrammes (say ½ ton) of pyrolignite of lime ; whilst, in the second case, pans taking a charge up to 1,500 kilogrammes (say 1½ tons) may be used. There is a manhole in the top of the pan, through which the pyrolignite of lime is fed. Above the decomposing pans is a floor, which serves as a reservoir for the acetate of lime received in sacks of 50 to 70 kilogrammes (110 to

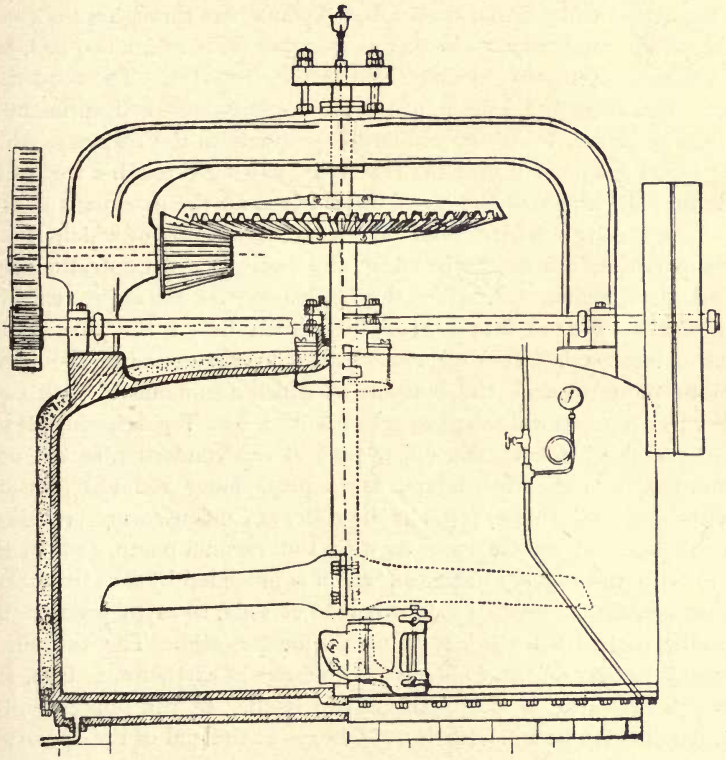

FIG. 31A.—Apparatus for the manufacture of acetic acid by Van der Linden's process, vertical section.

154 lb.). The pans are directly charged with pyrolignite, by shooting the contents of the sacks through openings in the floor, by the help of wooden hoppers. The pan being charged with acetate, the manhole is closed, and the agitator set in motion (Figs. 30 and 31) ; then, through a lead pipe, the sulphuric acid, contained in a lead-lined gauge tank, fixed and charged on the apparatus, is run in. The flow of sulphuric acid should be gentle, so as to get a very intimate mixture, and thus avoid

the formation of blocks, which might cause the mixer to stick. Soon after the sulphuric acid reaches the mass it begins to heat, the acetic acid begins to distil, even with a somewhat low fire, which is kept up for some time after having run in the sulphuric acid. Next to the pan is an apparatus called a dust compartment, 60 cm. wide by 1·3 metres deep (23·4 × 51·7 inches), placed on a floor at a level above the pan, so that the products which condense therein may fall back into the distilling apparatus. This dust chamber is made of lead, lined inside with large earthenware pipes, which protect the lead from the action of the acetic acid. From thence the vapours pass through a tin goose-neck to the condenser, consisting of a copper (or better, a tin) coil, laid in a wrought-iron tank, in which cold water circulates. The condensed acetic acid flows into a drum, of the same metal as the coil, surmounted by a pipe, which leads the condensed products to the chimney, whilst the liquid is siphoned into the reservoirs, passing through a sort of tin case through which all the liquid distillates from the pans pass, so that the liquid being distilled from each pan, the working of which has to be supervised, can be inspected at any moment. An operation lasts about seven hours. In the Van der Linden process, the acetic acid from the condenser flows into a glass flask, then into a tin pipe, which leads it into earthenware reservoirs similar to those used in the manufacture of nitric acid, the bottoms of which communicate with each other by a pipe, on which a gauge glass is fixed, by a T-piece, which is also in communication with the upper part of the condenser, as the condensation, like the distillation, takes place under reduced pressure. Two others of the upper pipes of the series of condensers are branched; on one pipe the branch connects with the vacuum pump, and on the other with the vacuum indicator, which is preceded by a short-necked bottle containing crystallised carbonate of soda, so as to preserve the metallic parts from the action of the acetic acid. The vacuum is brought to bear on the condenser by a series of earthenware taps, and through that also on the distilling pan itself. In the same way, the condensers may be isolated, if need be, as at the end of the operation, to re-establish atmospheric pressure inside the pan, or in the condensers when it is desired to extract the acetic acid. Between the earthenware condensers and the vacuum pump, scrubbers are fixed, consisting of at least two cast-iron columns 70 cm. (say 27½ inches) in diameter by 3 metres (say 10 feet) high (Fig. 32), in which are wooden or earthenware plates, supporting wood fibre moistened with a solution of carbonate of soda of 10° B. (15° Tw.), sp. gr. 1·075; that solution is introduced by the vacuum to the top part of the column, and as to the wash liquor from these scrubbers, as it contains acetate of soda, it is used in the

manufacture of that salt. As soon as all the sulphuric acid has been charged into the still, the disengagement of acetic acid slackens; the mass must therefore be heated for the acid to distil in a continuous stream, until the operation is terminated, a point which may be ascertained by the amount of acetic acid condensed, and by the flow from

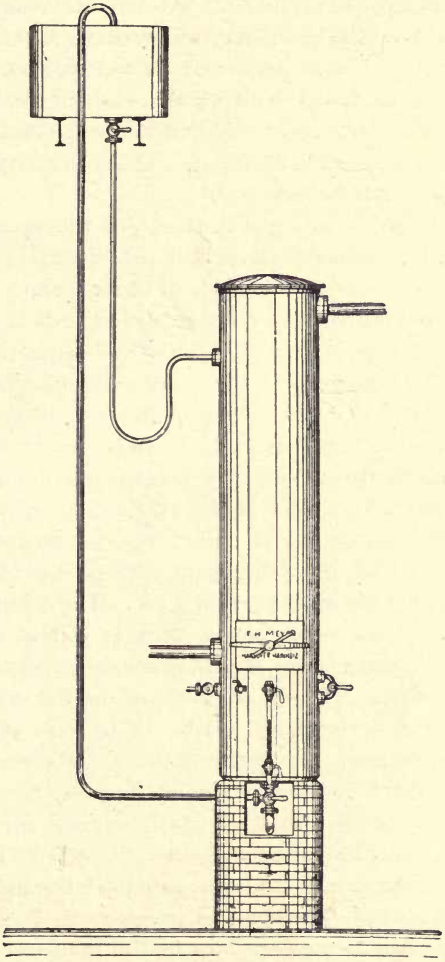

FIG. 32.—Wash column scrubber for the condensation of acetic acid vapours.

the condenser only dripping into the glass flask drop by drop. The fire is then banked up with ashes, steam is turned off, the manhole above the bottom of the apparatus is opened, still keeping the mixer at work, so as to run out the sulphate of lime into a gutter common to all the mixers, in which an archimedean screw revolves, which carries

the residues into a close chamber, where they cool for several hours, so as to avoid (especially near dwellings) the disengagement of bad smells due to empyreumatic products and to the acetic acid which remains in the free part of the apparatus as far as the condensers. The condensed acetic acid is propelled, either by a monte-jus, or by a rotary bronze pump, into different wooden reservoirs. In some of these it is brought, by the addition of water, to the commercial strengths (30° = 28 per cent. ; 40° = 38 per cent. ; 60° = 56 per cent. ; 70° = 65 per cent. ; 80° = 75 per cent. ; 85° = 80 per cent.—all approximately). From these reservoirs, the acetic acid is drawn into wooden casks for delivering to the different trades who utilise it. Other reservoirs are used to feed the acetic acid rectification plant.

Rectification of Acetic Acid.—The object of this operation is to produce acetic acid free from sulphuric acid and of a still greater strength than that obtained in the manufacture of acetic acid by sulphuric acid. The apparatus used is a column rectifier, similar to that used in alcohol distillation. It consists, in fact, of a horizontal copper pan (this metal protected from air being one of the least attackable by organic acids) of a capacity of 3,000 to 5,000 litres (say 660 to 1,100 gallons). One of the bottoms is readily removed, so that repairs can be easily executed, if need be, say to the steam coil by which the plant is heated. A vertical jacketed pan is also used in the rectification of acetic acid, but it requires to be constructed in quite a special manner, so as to be easily repaired. Its lid has a disengagement pipe in the centre and a manhole for cleaning the apparatus (Fig. 33). The column which forms the continuation of the copper pan is likewise of that metal, but with porcelain or earthenware plates, which, as must be understood, are more resistant than copper. The coil of the condenser is sometimes also of porcelain, but, as it is very fragile, it has to be fixed with the greatest of care. Cast-iron pans, the price of which is about the same as copper ones, are not to be recommended, because they are difficult to repair, and when worn out they are worth nothing, as compared with copper pans, which, if they are not usable, may still be sold by weight. As soon as the acid is run into the pan heat is applied slowly, so as to let such gases as sulphurous acid escape at the beginning. Then the steam in the coil is increased up to the moment when the acid vapours, passing into the column, begin to liquefy at the condenser. The heat applied to the pan and the water run on to the column, are then regulated by the strength of the acetic acid which it is desired to obtain. The first portions distilled contain the acetonic, methylic and sulphurous acid products, which are collected apart, and when the acetic acid, which condenses, titrates 96 per cent., it is collected in

the crystallisable acid reservoir. However, as the acetic acid obtained by this operation still contains empyreumatic products and traces of copper, if a more pure acid be required, it must be rectified once more, or it must be purified by the use of permanganate of lime in solution, which is made to act on the acetic acid contained in earthenware tanks. This treatment by permanganate is followed by fresh distillation in a copper pan, heated by a steam coil, the distilled products being condensed in an earthenware or in a silver coil, the latter being dearer and much less fragile. The residue from the rectification plant, of a

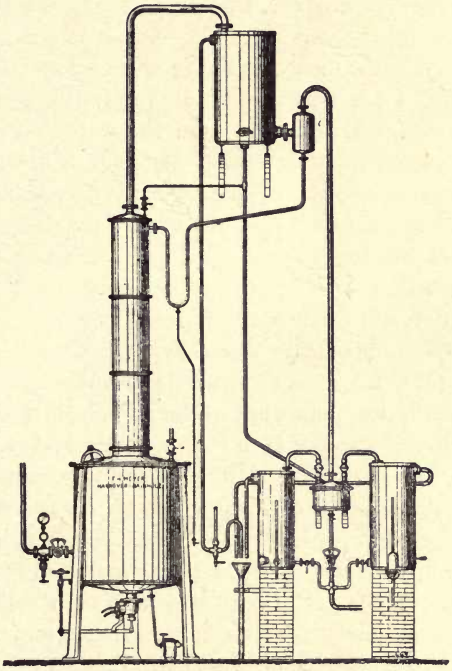

FIG. 33.—Rectifying column for acetic acid vapours.

more or less dark colour, consists in great part of acetic acid (along with which there occur the homologous acids contained in the acetate of lime) and tars ; these residues are led into a reservoir, where they are allowed to settle, so as to separate the acid which they still contain, which then undergoes further treatment. So as to prevent the entrance of too much air into the apparatus, the emptying of the rectifier should be done as rapidly as possible, without letting it cool too much before a fresh charge. In the installation of an acetic acid factory the wooden vats are located in a building adjacent to that in which the acetate of lime is decomposed and the acetic acid rectified ; the piping of these

vats is of tin or copper according to the quality of the acid conveyed therein. Finally, in winter care is taken so to heat this building that the crystallisable acid does not solidify. In addition to the different working departments, a building must be reserved for the engines, in which a dynamo for electric light is installed. An acetic acid factory, by the sulphuric acid process without a vacuum, producing about half commercial acid and half pure acid, expends about 250 kilogrammes (550 lb.) of coal per ton of pyrolignite of lime treated.

Glacial Acetic Acid.—Besides the crystallisable, *i.e.*, glacial, acid described in the previous chapter, this product may also be obtained by one of the following processes: (1) The oldest, but the least used at the present day, consists in decomposing fused acetate of soda by sulphuric acid (168° Tw.), then repeatedly crystallising and distilling the product so obtained, to free it from the water which it still contains; (2) the second process, termed Melsen's, is based on the property which neutral acetate of potash has, of combining with an equivalent of acetic acid to form a diacetate; that salt, when heated, abandons the water which it contains; it melts at about 148° C. (298·4° F.), giving off a little glacial acetic acid and boiling at 200° C. (392° F.) with decomposition and production of glacial acetic acid. During this distillation, as the temperature increases, care must be taken not to exceed 300° C. (572° F.), so as to avoid the formation of empyreumatic bodies. Practically the potassium acetate is crushed and introduced into a still, where an excess of 85 per cent. acetic acid is added to form the diacetate; the mixture is left to stand for some time and is then distilled. The vapours disengaged are then condensed in a silver coil. The first portions, which still contain too much water to be crystallisable, and which are used for the preparation of pure acetates, are collected apart; the acetic acid, which is then collected, is again distilled over neutral acetate of potash, then frozen and drained. By a final rectification, the acetic acid, which still dissolves essence of lemon (this product having the remarkable property of being slightly soluble in monohydrated acetic acid still containing some traces of water), is separated in a few minutes from the crystallisable (glacial) acetic acid.

Edible Acetic Acid (*Acide acétique bon goût*).—This product is obtained by means of acetate of soda and sulphuric acid 66° B. (168° Tw.), sp. gr. 1·840. One molecule of acetate of soda and two molecules of sulphuric acid are mixed in a copper still. This proportion yields bisulphate of soda as a residue, which can easily be run out of the still without fear of overheating, if naked fire heat be used, because the mass remains liquid until all the acetic acid is distilled. The soda salt used is that from the second crystallisation, simply passed through

the centrifugal in which it is washed. The sulphuric acid is run into the acetate of soda, and it is left to digest for some hours. The distillation is conducted over a naked fire or by steam at the atmospheric pressure, or *in vacuo* (Fig. 34); the concentrated acid distils first, whilst the water retained by the bisulphate of soda does not distil until towards the end, with the last traces of acid, which are collected apart for the preparation of certain acetates or for edible purposes. However, for this latter purpose the acetic acid must be purified from the impurities which it contains, such as hydrochloric acid or sulphuric acid, empyreumatic matters, acetate of copper, etc. For that purpose it is rectified in a steam-heated still (Fig. 35), over acetate of soda or acetate of lime, to retain mineral acids, or better, over red lead, which retains sulphurous acid as lead sulphate and decomposes the

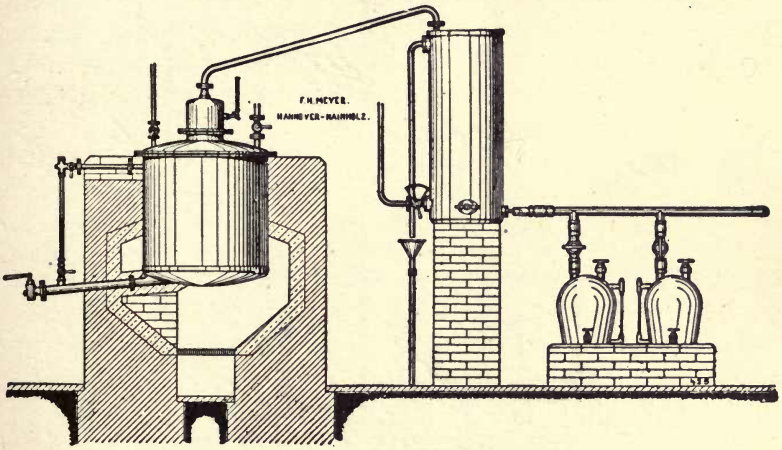

FIG. 34.—Plant for manufacturing edible acetic acid *bon goût* with distillation *in vacuo*.

empyreumatic matters by oxidation due to the peroxide of lead liberated by the acetic acid. The first products, collected in a silver condenser, being very weak, are intended for culinary uses; the strong acid which comes over afterwards is used in the preparation of the crystallisable acid. So as to correct the peculiar taste which acetic acid always retains, a small quantity of alcohol is added to that intended for edible purposes. The alcohol gradually etherifies and yields ethyl acetate, the more agreeable odour of which communicates a perfume to the acetic acid which causes it to resemble ordinary vinegar. Finally, when it is desired to produce a product quite exempt from foreign taste, as it should be for the preparation of table vinegar, the Mollerat process, which consists in preparing acetic acid by the cold method, is used.

Mollerat's Process.—One part of sulphuric acid is mixed with three parts of acetate of soda (in small white crystals obtained by crystallisa-

tion in motion) in a wooden vat fitted with a perforated false bottom and a wooden agitator. The mass is then allowed to stand for twelve hours, so as to give the sulphuric acid time to quite replace the acetic acid. The sulphate of soda, owing to its slight solubility in the cold, crystallises on the false bottom. The acetic acid is then separated by running it out through a tap below the false bottom, and when the sulphate of soda is well drained, it is mixed with a little water; afterwards a fresh quantity of acetic acid is drawn off, containing a little more sodium sulphate than the first, to which it is added. The acetic acid obtained is separated from the greater part of the sulphate of soda which it contains, by placing it to cool for eight to ten days in earthenware pots

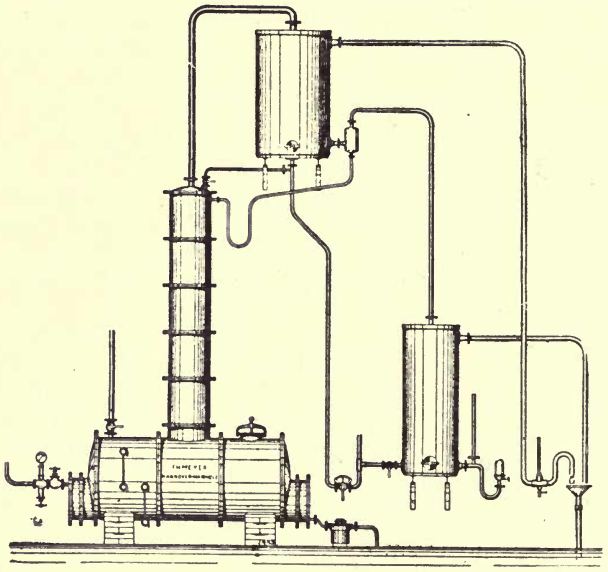

FIG. 35.—Plant for the manufacture of commercial acetic acid and chemically pure acetic acid.

of 50 litres (say 11 gallons) capacity, arranged in a tank round which water as cold as possible circulates. The acid is then decanted and completely purified by mixing it in a vat with a paste of pure acetate of lime, in sufficient amount to decompose the sulphate of soda which it contains; the sulphate of lime thus formed is gradually deposited. The clear acetic acid is finally drawn off and diluted to bring it to a suitable strength for consumption; it still retains traces of acetate of soda which have escaped the reaction, but that is no inconvenience. This acetic acid replaces vinegar in all cases where the taste of that obtained by distillation cannot be masked.

Anhydrous Acetic Acid.—The manufacture of this product belongs rather to the laboratory. Only a sketch of the different methods of preparing it will be given. (1) *By the action of oxychloride of phosphorus on acetate of potash.*—The oxychloride is run on to the fused acetate of potash drop by drop; the reaction is very energetic, though completely effected in the cold. Acetylic chloride is first produced, which, acting on the excess of acetate of potash, yields acetic anhydride—

$$3C_2H_3O_2K + POCl_3 = 3C_2H_3OCl + PO_4K_3$$
$$C_2H_3OCl + C_2H_3O_2K = KCl + (C_2H_3O)^2O$$

The acetic anhydride is distilled three or four times over acetate of potash, then the product is rectified alone, rejecting what passes over before 137·5° C. (2) *By the action of benzoyl chloride on acetate of potash.*—Potassium chloride and benzoyl acetate are first formed, but the latter, at the temperature at which this decomposition takes place, splits up into acetic anhydride and benzoic anhydride—

$$C_7H_5OCl + C_2H_3O_2K = KCl + \left.\begin{matrix}C_7H_5O\\C_2H_3O\end{matrix}\right\}O$$

$$2\left(\left.\begin{matrix}C_7H_5O\\C_2H_3O\end{matrix}\right\}O\right) = (C_2H_3O)^2O + (C_7H_5O)^2O$$

which are separated by distillation. (3) *By the action of oxychloride of carbon on acetate of soda.*—In a cast-iron cylinder, fitted with an agitator, pulverised acetate of soda is heated to about 140° C. (284° F.) without exceeding that temperature, because acetone might be produced, then a current of oxychloride of carbon is passed over the acetate of soda; acetic anhydride is disengaged which condenses and is afterwards subjected to fractional distillation.

Wood-Spirit. Methyl Alcohol.—The wood-spirit, as it is obtained after having separated it from the acetic acid in the distillation over lime, is brought to 25 to 30 alcoholometric degrees, by redistilling it in a pan heated by a coil. The vapours are liquefied in a condenser, and when the product which flows away falls below 25° G.L. it is run back into a phlegm reservoir until about half the volume contained in the pan has been distilled. The wood-spirit of 25° to 30° G.L. is then rectified over slaked lime, so as to bring it to 90° to 95° G.L. This product, according to its percentage of oils, is more or less miscible with water; in most cases it becomes turbid on the addition of water, because it contains neutral organic bodies or those which do not exhibit acid properties, the boiling point of which is in proximity to that of methyl alcohol itself. This alcohol, being too impure for the great majority of commercial purposes, is redistilled, so as to obtain a methylic alcohol just pure enough for the purposes for which it is to be used in industry.

The apparatus, still somewhat used in certain factories, the description of which is borrowed from Ch. Vincent's *Carbonisation des Bois en Vase Clos*, consists of three distinct parts: (1) of a copper or wrought-iron pan of 2,000 to 6,000 litres (say 440 to 1,320 gallons) heated on a naked fire (Fig. 36), or by a steam coil (Fig. 37). (2) Of the rectifying column, which consists of three lenticular plates (Fig. 36), or a copper receiver, in which the goose-neck terminates (Fig. 37), surmounted by six trunks of the same shape supporting lenticular plates, which compel the vapours to pass along the sides of each of the trunks connected between each other by a central pipe. The top of each trunk is in the form of a basin, in which water brought by a tap, the flow of which is regulated on the top plate, circulates; the water afterwards flows on to each

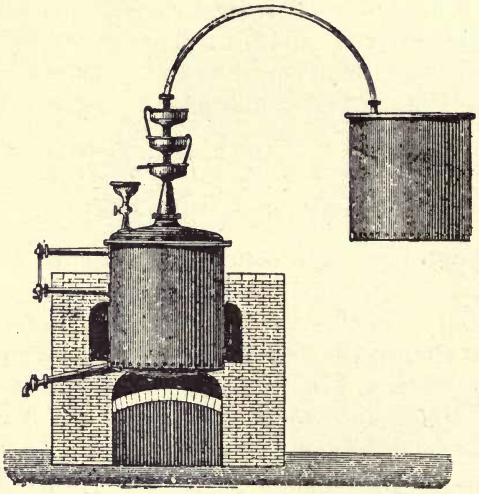

Fig. 36.—Still for the distillation by naked fire of wood-spirit for French excise purposes (*des méthylènes Régies*).

of the lower plates through a small pipe running from the bottom of the basin; from the bottom of the lower receiver a pipe starts which returns the products condensed in the rectifier to the pan. (3) Of a coil to condense the vapours issuing from the apparatus. In contact with the multiple cooling surfaces of the column, the mixture of alcoholic and aqueous vapours is cooled. Owing to difference in boiling points there is a more abundant condensation of aqueous vapours than of alcoholic vapours, which causes an increase in the alcoholic strength of the vapour, as occurs in the glass column in the laboratory. In fact, the liquid condensed in the top of the column, having a temperature of about 66° C. (150·8° F.), approaching the boiling point of methylic

alcohol, meets as it descends the current of vapour from the pan, which in its turn volatilises the alcohol condensed by the water in the top plates, whilst at the same time it reduces a corresponding amount of water to the liquid state. It follows that with a column of six trunks of 50 cm. (say 19·5 inches) in diameter, 95 per cent. alcohol can be produced in one operation, starting with 30 per cent. phlegms. In addition to methyl alcohol, the product thus obtained contains acetone, ethyl aldehyde, formic aldehyde, allyl alcohol, a small quantity of methyl acetic ether (methyl acetate) which has escaped

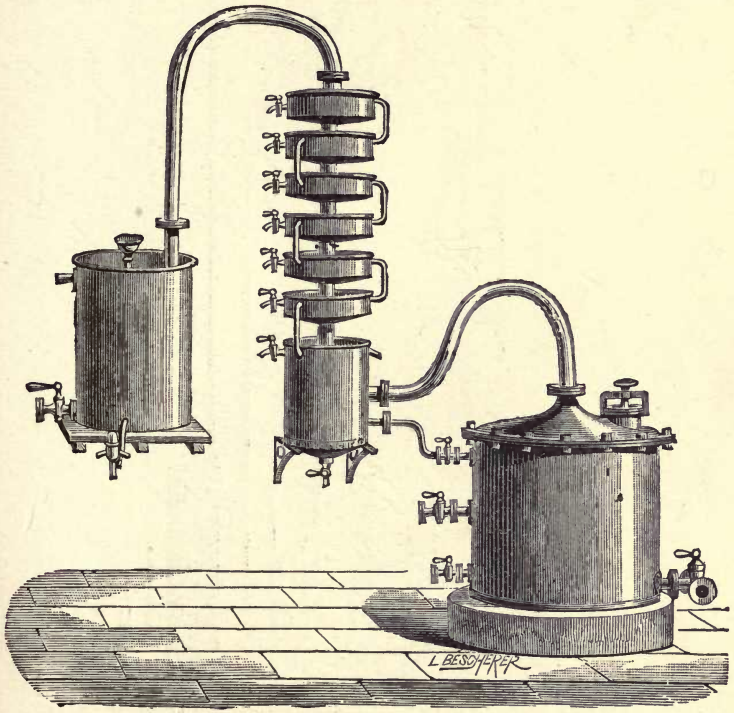

Fig. 37.—Steam-heated still for the distillation of wood-spirit.

the slaked lime and certain hydrocarbides, which cause it to become milky when water is added to it. With these impurities it suits perfectly for certain uses, such as the denaturation of ordinary alcohol (for burning, etc.), the manufacture of different varnishes, etc.; but when it is to be used for the preparation of certain aniline colours, it must be purified by a further rectification in more complete plant, so as to free it from those hydrocarbides which, when oxidised during the course of manufacture, would yield black products.

To purify such alcohol it is first diluted with distilled water so as to bring it to 50 per cent. strength; after standing a few days, the liquid forms two layers, the surface one of which contains the major portion of the hydrocarbides. The lower layer is drawn off and rectified with 2 to 3 per cent. of lime in the preceding apparatus, or in a column rectifier followed by a dephlegmator, with return to the top part of the column (Fig. 38). At the commencement of the rectification every effort is made to obtain the greater part of the acetone,

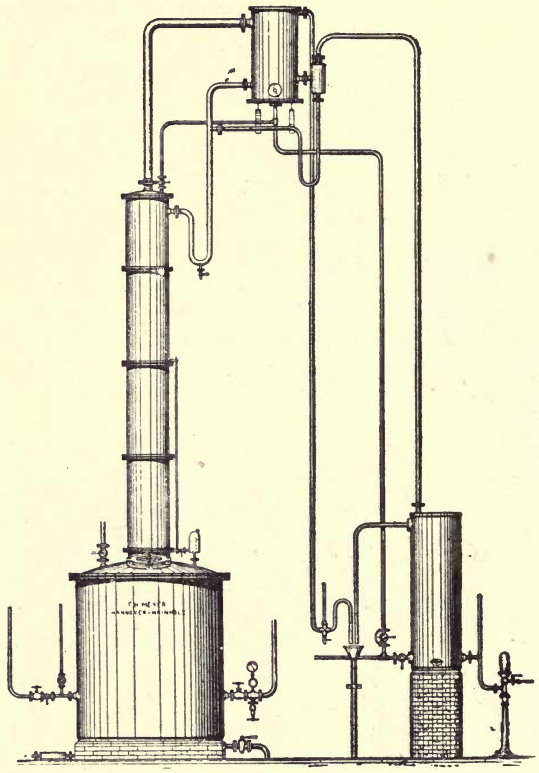

FIG. 38.—Steam-heated still for the distillation of wood-spirit, surmounted by rectification column.

then a slight current of water is run into the column, which is still kept comparatively warm, and the condensed products are fractionated, collecting them in separate reservoirs, from which they are afterwards drawn off and more pure products extracted from them. The condensed liquids may be divided into: (1) First runnings, testing 60 down to 8 per cent. of acetone. (2) Middle runnings, miscible with water containing 7 down to 1 per cent. of acetone. (3) Middle run-

nings, which do not give limpid solutions with water. (4) Portions containing allylic alcohol. (5) Last runnings, containing oils. The distilled products are tested, from an acetone point of view, by means of soda, by adding 20 c.cm. of a solution of caustic soda of density 1·3 to 10 c.cm. of alcohol contained in a graduated tube, and agitating strongly. It is then allowed to stand, when the acetone separates entirely in a supernatant layer; this test gives no result unless the alcohol holds 1 per cent. of acetone in solution. The distillation is

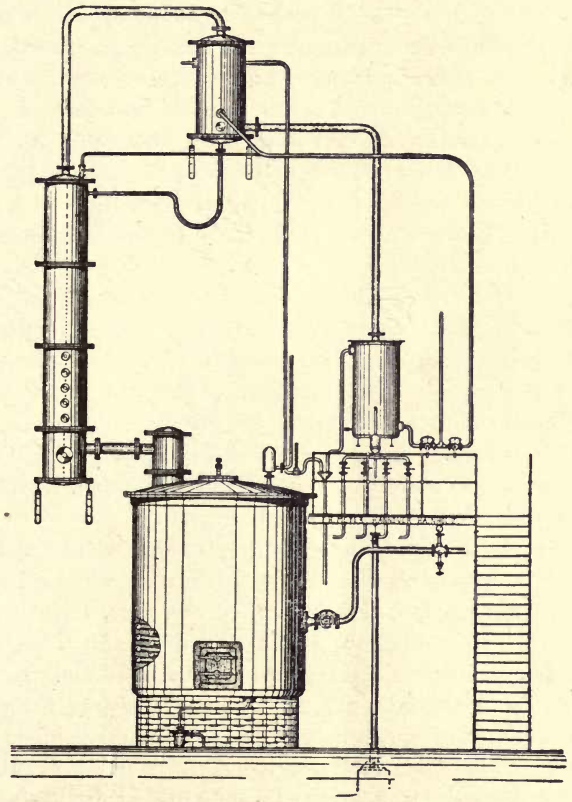

Fig. 39.—Steam-heated still, with independent rectification column, for rectification of wood-spirit.

still followed up, testing the miscibility of the alcohol with water, and taking its strength from time to time by the alcoholometer. At the end of the operation, the distillate is no longer miscible with water; the last portions have a milky appearance, and are merely an emulsion of oil and water. The column apparatus (Fig. 38) may be advantageously replaced by a rectifier, with arrangements for the easy cleaning of the still and the column (Fig. 39), this latter not being mounted directly

on the still. In spite of all the precautions which may be brought to bear in this second rectification, the methyl alcohol obtained is not yet sufficiently pure for the manufacture of methyl aniline, for example, or for the preparation of methylic ethers in the laboratory. It then undergoes a third rectification in presence of a small quantity of sulphuric acid, and the distillate, between the temperatures 64° and 67° C. (147·2° to 152·6° F.), is collected for the manufacture of the above-mentioned bodies. Finally, when it is desired to obtain methyl alcohol free from acetone, the *Rotten* process is adopted, which consists in combining acetone with a halogen. For this purpose, methyl alcohol is distilled in a reflux condenser, into which a current of gaseous chlorine is passed, which, inert to the alcohol, transforms the acetone into chlorinated compounds, of boiling point about 120° C. (248° F.), much higher than that of methylic alcohol; the product is then distilled, after which the alcohol is rectified over lime to remove the last traces of chlorine. According to the inventor, this process yields methyl alcohol perfectly free from acetone.

Continuous Distillation.—As often, for commercial reasons, it is more profitable for a wood charcoal factory to produce methyl alcohol containing small proportions of acetone than alcohol free from acetone, a sketch is here given of some rectification plant, in which the distillation is so much the more interesting because it is continuous, and that is an important step in advance in distillery progress. Continuous rectification enables more pure alcohols to be produced, whilst a considerable saving is effected at the same time.

Coupier's Continuous Rectifier.—In this rectifier, the methylic vapours, after traversing a series of plates (through which they bubble) in the rectifying column, pass into three receivers, termed analysers, arranged as shown in Fig. 40, at different depths in a tank of water at a given temperature. The vapours pass into each analyser through a dipping tube in the form of a rose, and bubble into the condensed liquid, the overflow of which is brought back by independent pipes on to certain of the bubbling plates on the rectifier, making that from the last analyser discharge at a higher level than that from the first. A tap, placed under each analyser, enables the progress of the distillation to be followed by taking samples from it from time to time.

Barbet's Rectification System.—In Barbet's system, the object of rectification is to make three assortments of the substances contained in the crude alcohol. (1) The aggregate of all which is more volatile than alcohol, or the first runnings. (2) Pure alcohol. (3) The aggregate of all which is less volatile than alcohol, or the last runnings. The crude alcohol is freed in a preliminary operation from all products

more volatile than alcohol—that is *continuous purification*. The purified alcohol is sent to the continuous rectifier properly so called, which brings it to 96 and 97 per cent. and eliminates the last running's products. Barbet's continuous rectifier (Fig. 41) comprises two quite distinct organs: the purifier and the rectifier. The purifier is an ordinary distilling column, A, surmounted by a small column, furnished with plates for the concentration of the ethers and to retain the pure alcohol. The alcohol, to be purified, flows into the forewarmer, R, by the pipe, S; there it is heated by the exhausted liquors issuing from the column, G. A notable amount of fuel is thus saved; the alcohol passes into the upper part of the column, A, and flows from plate to

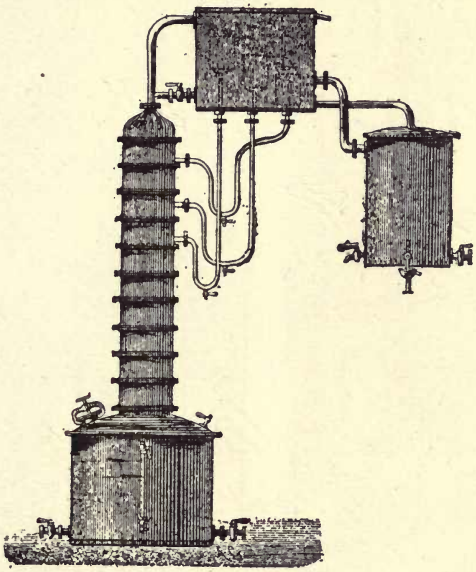

Fig. 40.—Continuous still for distillation of wood-spirit.

plate until it reaches the lower part, which is heated by a coil. It is provided with a steam regulator, which is to be seen adjacent to column A. The vapours which rise from the bottom of this column consist of acetone, ethers and alcohol; as they successively pass through all the liquids spread on the plates, which become gradually less and less hot and more and more rich in acetone and in ethers, these vapours are deprived of their alcohol, and enriched with products of lower boiling point. These products of lower boiling point are concentrated in the small column and pass to the condenser, B, where a retrogradation produces a methodical enrichment of these ethers and

of the acetone, which are finally liquefied in the refrigerator, C, and flow out by the test glass, P.

The alcohol freed from first runnings passes by the pipe, N, into

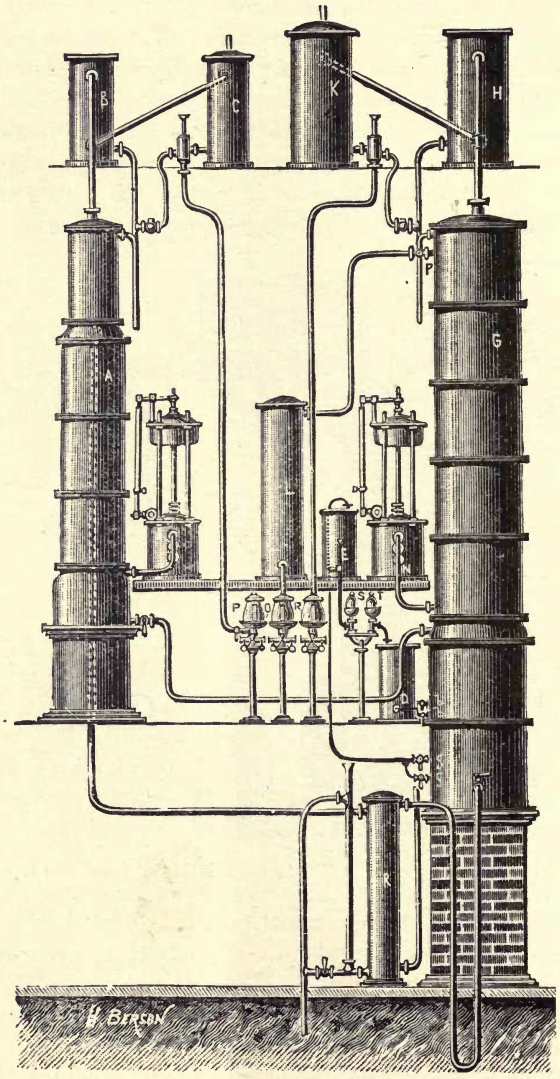

FIG. 41.—Barbet's continuous rectification plant for wood-spirit and acetone.

the rectifier, G, which is an ordinary distilling column, consisting of two kinds of plates: (1) exhaustion plates in the lower part, (2) rectification plates above the former. The purified alcohol, in the

upper part of the exhaustion plates, descends from plate to plate and reaches the bottom of the column, which is heated by a steam coil provided with a steam regulator. During this passage it is deprived of all its alcohol; the exhausted water flows away by the siphon, passes through the forewarmer, R, where it cedes a portion of its caloric to the crude alcohol to be purified and then runs down the drain. The alcoholic vapours become stronger and stronger as they traverse the rectification plates. They reach the condenser, H, where a retrogradation sends back those vapours which condense there to the upper part of the column, G. The pure alcoholic vapours, which traverse and wash themselves in the condenser, H, pass to the refrigerator, K,

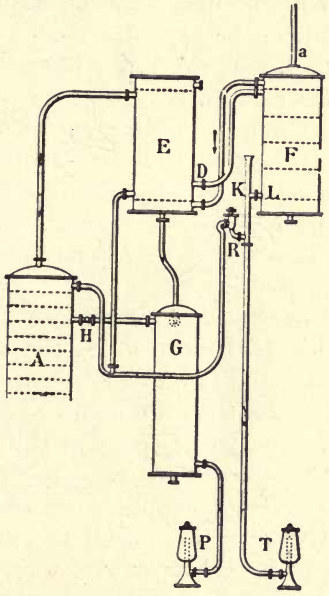

FIG. 42.—Diagram to illustrate regulation of Barbet's continuous rectifier.

and the resultant liquid, that is to say pure alcohol, flows out through the test glass, Q. The classification of the products is effected on the plates in the order of their purity, and the only function of the condenser, H, is to provide a wash water in which the alcoholic vapour refines itself. Thus to have a pure alcohol it is necessary that the washing alcohol be perfectly pure. Suppose, says Barbet, the alcohol from the test glass contains a small proportion of first runnings, the retrogradation will contain the same impurity, and will reintroduce it on to the top plate of the column. But the most volatile products are re-evaporated first, and there will remain on the top plate vapours

in a state of purity. The phenomenon of purification is surprisingly energetic on the top plate. The small refrigerator, E, serves to determine the exhaustion of the residual waters; a thin stream of these waters flows constantly from the test glass, S, where they can be examined at any moment. The regulation (Fig. 42) of the flow in the continuous rectifier is rendered invariable by giving to the refrigerator, F, the same height as the condenser, E, and by placing them both on the same level. The cool alcohol issues by the pipe, L, fitted with an air pipe, K, and descends to the test glass, T. The alcohol drawn off from the top part of the column, A, at H is cooled in G, and flows out through P; on the descending alcohol pipe is branched a valve, R, connected with the retrogradation from the condenser, E. If the tap (near T) be closed whilst the apparatus is at work, the alcohol will ascend in the tube, KN, raise the valve, R, and mix with the retrogradation to enter the rectifier, A (at top). Without perturbation of any sort, without requiring more water than formerly, the apparatus continues to rectify at the same rate even though there is no outflow of alcohol. By opening the tap, N, more or less, the tap, H, being open more or less, pure alcohol is obtained. The flow of the two test glasses, P and T, can thus be regulated, and always in an invariable manner.

Lepage & Co. of Paris construct a sort of rectifying still head or capital, which not only has the advantage of fitting any still, but is also economical of space, by doing away with the goose-necks and condensers of ordinary stills. The rectifying still head realises a good use of the condensing water, which may be replaced by the liquid to be distilled when it is desired to distil continuously. The alcoholic vapours from the pan traverse different rectifying plates contained in the chamber, C (Fig. 43). These plates, having the form of lenses with a double envelope, are cooled by a current of cold water, *r*, flowing above the funnel, E, regulated at will by a tap, by which the rectification, which may be suppressed at any given moment, may be varied as desired. The alcoholic vapours afterwards penetrate into a double coil, S, where they circulate in an inverse direction to a current of cold water brought by the pipe, D; the outflow of the hot water, *s*, having done duty in the rectification rejoins that (*s'*) from the condensation by the pipe, F, for their common discharge. In continuous distillation without the aid of water the liquid to be distilled first passes through the condensing coil (Fig. 44), then on to the rectifying plates. The liquid, after having been distilled in the column, falls exhausted into the pan, the residue from which is evacuated by a siphon, I. The alcohol flows from the test glass, E, at a constantly uniform strength determined by the feed tap, R.

Preparation of Pure Methyl Alcohol.—Wood-spirit, as obtained by one of the processes given above, is not chemically pure, for it still contains 0·4 to 0·5 per cent. of acetone, aldehyde, methyl acetate, which can only be separated with difficulty by a series of fractionations. Other methods of purification besides Rotten's must therefore be resorted to, especially in laboratories, in which advantage is taken of, at one time, the properties of methyl alcohol, at other times, of those of acetone. In one of these methods, methyl alcohol is combined with calcium

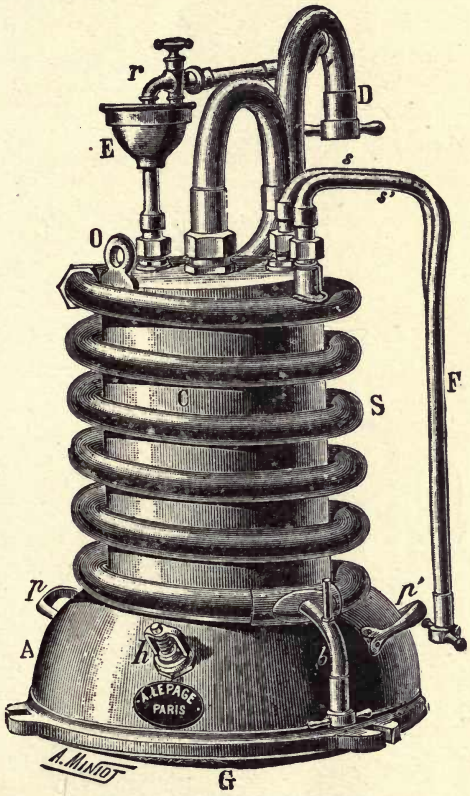

Fig. 43.—Rectifying still head.

chloride in powder. The crystals obtained are, after draining, heated on the water bath to drive off the acetone and other volatile uncombined products, then they are taken up by a certain amount of water, which breaks up the compound, and the methyl alcohol is distilled. Another special process for eliminating acetone consists in combining the latter with a freshly prepared concentrated solution of sodium bisulphite. The whole is allowed to stand while the crystals separate out; then the

clear liquid is distilled. The distillate is methyl alcohol free from acetone. The acetone may be converted into chloroform or into iodoform by treating the methyl alcohol by a milk of lime and a concentrated milk of chloride of lime; by afterwards submitting the chloroformic liquor thus obtained to distillation, the chloroform passes over first; it

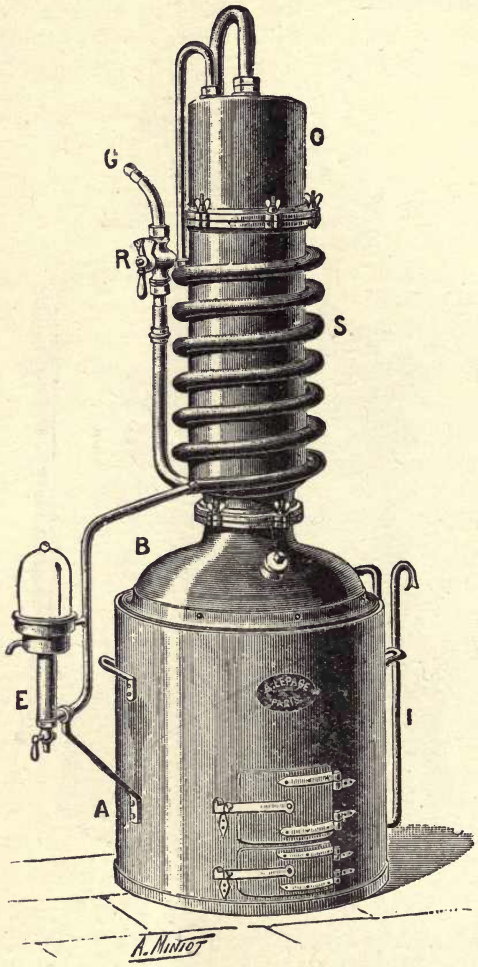

FIG. 44.—Lepage's rectifier for continuous distillation.

is collected under water so as not to lose methyl alcohol (the waters are used up in the manufacture), then, when pure methyl alcohol distils, it is collected apart.

Preparation and Properties of the Acetates. Different Uses of these Salts. Neutral Acetate of Potash.—This salt ($C_2H_3O_2K$) is prepared by directly

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neutralising a solution of acetate of potash or of carbonate of potash by acetic acid. The solution, evaporated to about 37° B., sp. gr. 1·345, is set to crystallise in wooden tanks lined with lead; the crystals obtained, silky and deliquescent, are then centrifuged to separate the mother liquors. The aqueous solution of acetate of potash, evaporated to dryness, is transformed into a lamellar mass with a very brilliant fracture termed *terre folière du tartre des anciens* (*terra foliata tartari* and *febrifuge salt of Sylvius*); it melts at about 292° C., yielding foliated deliquescent crystals on cooling. It is used as an energetic dehydrating and decomposing agent. A solution evaporated with excess of acetic acid gives a crystalline mass of acid potassium acetate $C_2H_3O_2K_1C_2H_3O_2H$ melting about 148° C. (298·4° F.), and is used, as already mentioned, for the manufacture of glacial acetic acid.

TABLE IX.—GIVING THE PERCENTAGE OF ANHYDROUS ACETATE OF POTASH IN AQUEOUS SOLUTIONS OF THE SALT OF DIFFERENT DENSITIES

Densities at 15° C. (59° F.).	Potassium Acetate, per cent.	Densities at 15° C. (59° F.).	Potassium Acetate, per cent.
1·049	10	1·2105	40
1·1005	20	1·2685	50
1·1545	30	1·3285	60

Acetate of Soda ($C_2H_3O_2Na + 3H_2O$), *also termed terra foliata mineralis* (*terre foliée minerale*).—The pyroligneous acid, separated by distillation from the methylated products, is run into wide, shallow, wooden or enamelled iron tanks, which are only filled two-thirds; Solvay soda ash is added gradually, taking care to wait a few minutes, after each fresh addition of alkali, to allow the carbonic acid to escape and to let the effervescence calm down so that the liquid does not overflow. In proportion as the solution becomes less and less acid, the tarry matters held in solution, as already mentioned, by the acetic acid, separate and float on the liquid, and when the liquid is neutralised, which is the case when a fresh addition of soda produces no more effervescence, the whole is allowed to stand for some time for the tars to aggregate; these are skimmed off and the solution of acetate of soda is sent to the evaporation plant. If the waste heat from the charcoal furnaces is to be used for concentrating the acetate of soda, rectangular wrought-iron tanks are laid over flues, into which the furnace gases can be made to pass by means of a damper before going to the chimney. The saline solution is led into the tank the most distant from the furnace, then into the next to balance the evaporation, in proportion as it is produced, until the liquid of the last marks 27° B., sp. gr. 1·23. The damper on the flue is

then momentarily closed and the gases pass directly to the chimney, until the contents of the tank, brought to the right degree of concentration, are emptied into the wrought-iron crystallisers. This empty tank is now filled with the contents of the preceding one, and the evaporation is continued by bringing the furnace gases once more under the tanks. As, during this operation, tar continues to float to the surface of the mass and to soil the product, care must be taken to skim it off as soon as formed. The crystallisation of acetate of soda takes several days; when it is terminated, an orifice is opened at the base of each crystalliser. The latter being slightly inclined, the mother liquors flow into a gutter which

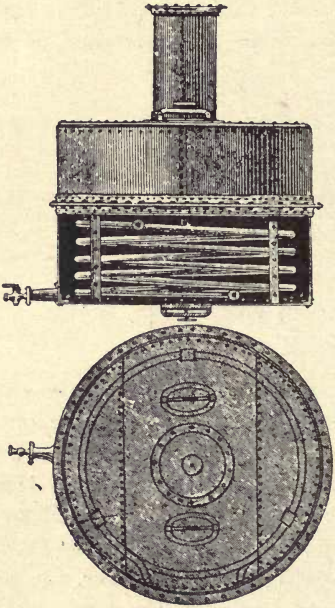

Fig. 45.—Evaporation pan for evaporating acetate of soda liquors.

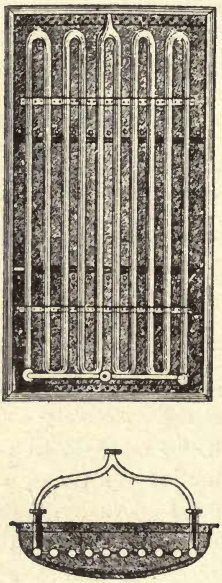

Fig. 46.—Concentration pan for acetate of soda liquors.

leads them to a tank at a lower level. The salt obtained in this way is placed, after draining, in a wrought-iron or cast-iron pan (Figs. 45, 46), and the requisite amount of water added to make a boiling solution of 27° B., sp. gr. 1·23, which is afterwards set to crystallise in tanks. After six to ten days crystals of acetate of soda are obtained larger than the preceding, but still coloured brown; this acetate of soda of the second crystallisation may be bleached by centrifuging it and washing it in the centrifugal with a solution of purified acetate of soda. To prepare white acetate of soda, the second crystallisation crystals are melted, without addition of water, in a cast-iron pan, then the product is passed, by small

quantities, into an adjacent pan termed the *frit*, fitted with a lid and a mechanical agitator (Fig. 47). The acetate of soda, brought to about 380° C. (656° F.), first dries, then melts, and the tarry matters decompose and volatilise. After an hour and a half the fused salt is, according to the installation, lifted by an iron dipper (*poche en fer*) and run into an iron gutter, which leads it into a cast-iron or wrought-iron pan, which is shut up so as to avoid projections on the outside, and containing water, in which it dissolves. The fritted acetate of soda may also be run by a free discharge on to plates, where it solidifies; it is then dissolved in a vat, fitted with a perforated steam coil and containing the amount of water necessary to make a solution of 20° to 22° B. (32° to 36° Tw.),

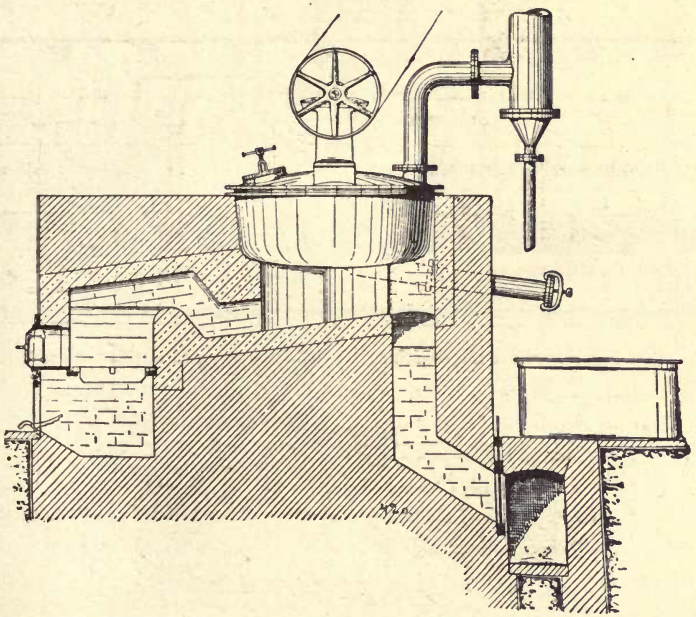

FIG. 47.—Plant for drying and fusing acetate of soda crystals.

sp. gr. 1·16 to 1·18. This new solution, after standing and decantation, is filtered through animal charcoal, so as to decolorise it as far as possible. When the fritting is well done, filtration through animal charcoal may be dispensed with; it is simply passed to the filter presses (Fig. 48) to separate the molecules of charcoal which it holds in suspension and originating from the decomposition of organic matter. The solution is evaporated to 25° B. (42° Tw.), sp. gr. 1·21, then it is run into flat circular copper vessels 1·8 metres (say 6 feet) in diameter by 25 cm. (say 10 inches) deep (Fig. 49), fitted with a mechanical agitator, so as to obtain small crystals more pure and thus more easily centrifuged and washed

by a solution of acetate of soda. The salt obtained in this way is used to prepare acetic acid by the Mollerat process. The fritting of acetate of soda from the second crystallisation being rather a delicate operation, owing to the temperature to which it must be submitted without decomposing it, it is preferred in certain factories to suppress this operation and to prepare a boiling solution of acetate of soda, of 15° to 16° B., sp. gr. 1·115 to 1·125, which is slowly passed through granular animal charcoal filters similar to those used in sugar works (Fig. 50). Liquors

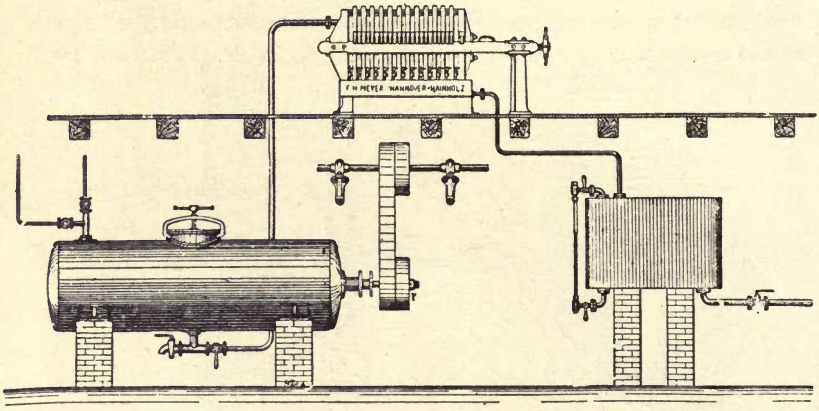

Fig. 48.—Plant for the solution and filtration of fused acetate of soda.

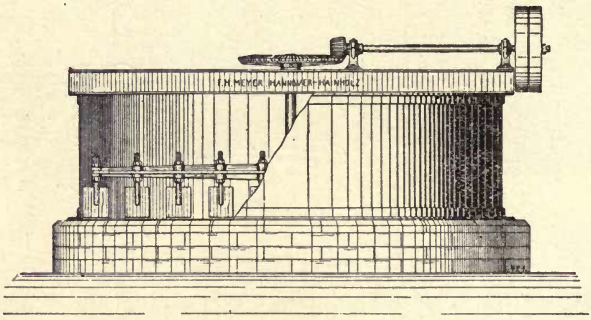

Fig. 49.—Plant for the crystallisation in motion of acetate of soda.

are thus obtained sufficiently decolorised to yield, after concentration and crystallisation, blonde crystals of acetate of soda as pure as those prepared by the preceding process. This method of purifying requires considerable quantities of animal charcoal.

Hanriot's Process.—The solution of the first crystals is treated by tannin or crushed defibred oak or chestnut wood ; the tannin precipitates the tarry matter from the liquors, which are then separated by decanta-

tion or filtration. The solution, after evaporation and crystallisation, yields acetate of soda, suitable for the manufacture of edible acetic acid (*acide acétique bon goût*). Finally, by neutralising with Solvay soda ash, the acetic acid, obtained by decomposing pyrolignite of lime by a mineral acid, and crystallising, white acetate of soda is obtained sufficiently pure to be used, say, in dyeing. Neutralisation is effected in wooden or in enamelled iron vats containing, say, 70 per cent. acetic acid, to which, in small portions, is added almost the theoretical amount of Solvay soda ash, because it is necessary to bear in mind that a small amount of acetic acid is entrained by the carbonic acid given off during the reaction. As soon as the operation is finished, the product is dissolved in water, or,

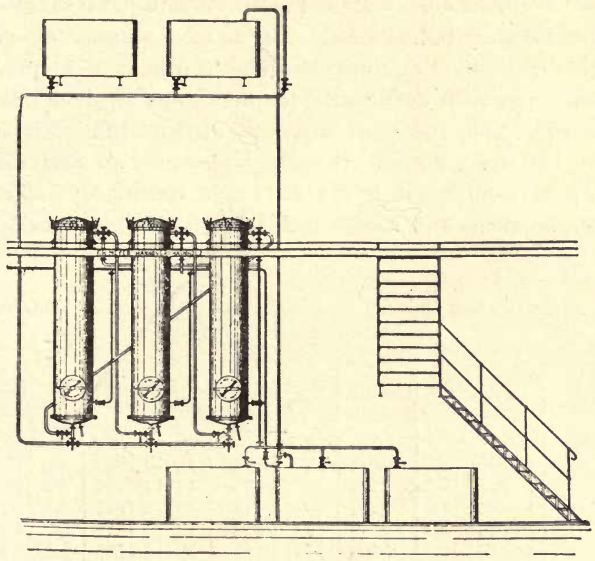

FIG. 50.—Animal charcoal filters for decolorising acetate of soda solutions.

better still, in the acetate of soda mother liquors from a previous crystallisation, previously heated in a copper or cast-iron pan fitted with a lead coil. Care is taken to keep the solution slightly alkaline so as not to dissolve the metal of the pan. The acetate of soda is run into the pan in small quantities at a time so as to allow the carbonic acid remaining in the saline mass to escape without the solution overflowing. When all is dissolved it is heated to the first bubble and the boiling solution is brought to a density of 27° B., sp. gr. 1·23. It is allowed to stand for several hours, then the clear portion is siphoned off and placed to crystallise in rectangular vats of 25 cm. (say 10 inches) deep, where a little acetic acid is added to it, whilst it is still very hot, to neutralise its

slight alkalinity. After two or three days, according to the season, the crystallised acetate of soda is separated from the mother liquor, then passed through a centrifugal with direct motor, such style of driving enabling that machine to be installed in any building through which steam passes. After being centrifuged the acetate of soda is bagged up in 2 cwt. bags and put on the market. Acetate of soda ($C_2H_3ONa + 3H_2O$) is efflorescent in dry air; it melts in its water of crystallisation, which it gradually and then completely loses as it is heated up to 319° C. (606·2° F.).

Acetate of Ammonia.—This salt is prepared by passing ammonia gas through glacial acetic acid until no more is absorbed. The liquid becomes hot by the combination of the two bodies, then on cooling it is converted into a crystalline mass. The aqueous solution of ammonium acetate, likewise called *Spirit of Minderus,* can likewise be prepared by mixing ammonia with acetic acid in proportion of about 30 parts of ammonia at 22° for 100 parts of acetic acid at 40°. That solution, heated to boiling, gives off ammonia and yields an acid salt which crystallises in needles, which, if the heat be continued, is decomposed into water, ammonia and acetic acid, to finally yield acetamide which distils.

TABLE X.—SHOWING THE DENSITY OF SOLUTIONS OF ACETATE OF LIME OF VARIOUS STRENGTHS AT 15° C. (59° F.)

Density.	Per cent.	Density.	Per cent.	Density.	Per cent.	Density.	Per cent.	Density.	Per cent.	Density.	Per cent.
1·0066	1	1·0362	6	1·0527	11	1·0708	16	1·0925	21	1·1189	26
1·0132	2	1·0394	7	1·0562	12	1·0750	17	1·0996	22	1·1248	27
1·0198	3	1·0426	8	1·0597	13	1·0792	18	1·1027	23	1·1307	28
1·0264	4	1·0458	9	1·0632	14	1·0834	19	1·1078	24	1·1366	29
1·0330	5	1·0492	10	1·0666	15	1·0874	20	1·1130	25	1·1426	30

White Acetate of Lime.—White acetate of lime is prepared by saturating lime with acetic acid: one part of slaked lime and three parts of 60 per cent. acetic acid. The milk of lime is prepared and added by small quantities to the acetic acid, taking care that the temperature does not rise too much, so as to have the smallest possible loss of acetic acid. The neutralisation finished, the mass is allowed to cool, and the white mass so obtained is centrifuged. The acetate of lime is then laid to dry on enamelled iron plates heated, for example, by waste furnace gases.

Acetate of Alumina (Calico Printers' Red Mordant).—A solution of this salt which does not crystallise is used as an ordinary mordant in calico

printing. It is prepared as a more or less dilute solution, according to the purpose for which it is intended, either by double decomposition of a soluble acetate and an aluminium salt or by solution in acetic acid of freshly precipitated alumina. By double decomposition acetate of lead or acetate of lime is used, or even, and preferably, acetate of baryta when it is desired with sulphate of alumina to obtain an acetate exempt from sulphuric acid; sulphate of lead being slightly soluble in an acetate and sulphate of lime in water. Acetate of alumina having little stability, especially when hot, heat decomposes it, liberating acetic acid; it should therefore be made cold. If to be made from lead acetate, a solution of that salt, of 45° B. (91° Tw.), sp. gr. 1·455, is prepared, which is mixed cold with a solution of sulphate of alumina of 22° B. (36° Tw.), sp. gr. 1·180. The whole is allowed to settle and the solution of acetate of alumina is decanted. Acetate of alumina prepared from acetate of lime is made by running in a solution in molecular proportion of acetate of lime—by small quantities at a time, to avoid an increase of temperature—into a molecular solution of sulphate of alumina. After having mixed the mass well it is allowed to stand, then decanted, and, if it be desired that this solution contain no sulphate, the operation is finished by the addition of a small quantity of acetate of baryta. Acetate of alumina suitable for dyers is prepared from alumina freshly precipitated from an aqueous solution of potash-alum, or ammonia-alum, by carbonate of soda. The gelatinous alumina is dissolved in acetic acid. By adding increasing quantities of an alkaline carbonate to a solution of the normal salt, solutions of different basic acetates may be obtained. Impure solutions of acetate of alumina, prepared by precipitating an alkaline solution of an alum by lead acetate, are often met with in commerce. For this purpose 70 lb. of alum are dissolved in 5 gallons of water. Then 100 lb. of acetate of lead in fine powder are gradually added with constant stirring; sulphate of lead is formed which is deposited. The liquor contains, besides acetate of alumina, sulphate of potash, sulphate of ammonia, or sulphate of soda, according to the nature of the alum used, and traces of sulphate of lead. The trade term applied to the different acetates and sulpho-acetates of alumina used in the industry is that of *red mordant*, because they are universally used, in dyeing and calico printing to produce alizarine reds on cotton.

Acetate of Iron.—The acetate of iron used in dyeing black is made from pyroligneous acid freed from methyl alcohol and the greater part of its tar. It is poured, boiling, on iron turnings in wooden vats; the iron is soon attacked with disengagement of hydrogen, whilst the tars, held in solution by the free acetic acid, rise to the surface of the liquid

in a thick layer which is skimmed off. After twenty-four hours the pyroligneous acid, converted into a mixture of acetate of protoxide of iron (ferrous acetate) and of sesquioxide of iron (ferric acetate), is withdrawn ; it marks 14° B. (22° Tw.), sp. gr. 1·100. As to the iron which remains in the vat, it must be taken out to revivify it. For that purpose it is piled in heaps and set on fire to burn off the tars which cover the undissolved turnings. The iron is then passed through a sieve to separate the oxide which is formed during the combustion, after which it can be used for a fresh operation.

The acetate of protoxide of iron (*ferrous acetate*), used in making certain inks and in calico printing, is prepared by dissolving iron in acetic acid in a copper pan. To facilitate the reaction the whole is heated to about 70° to 80° C. (158° to 176° F.), then it is allowed to cool. As the oxygen of the air transforms this salt into the acetate of sesquioxide of iron (ferric acetate) it is run off into closed vessels. The presence of catechol, which acts as a reducer in acetate of protoxide of iron (ferrous acetate), prevents it from oxidising too rapidly, a fact which enables it to be preserved. It may also be prepared by double decomposition of a solution of acetate of lime or of lead and ferrous sulphate (green vitriol).

Ferric Acetate.—In order to prepare ferric acetate, either a solution of ferric sulphate may be decomposed by an acetate, or the more commercial process may be adopted of manufacturing it by abandoning iron turnings immersed in acetic acid to the prolonged action of the air. The operation, which lasts several weeks, is carried out in casks fitted with a false bottom ; the liquid which flows out at the bottom is repassed through the cask from time to time over the mass being treated. Finally, when the solution marks 10° B. (15° Tw.), sp. gr. 1·075, by the areometer, it is concentrated over a naked fire to 15° B., sp. gr. 1·115, for delivery to purchasers.

Zinc Acetate.—This salt is obtained by dissolving zinc, carbonate of zinc or oxide of zinc in acetic acid. With zinc oxide a milky paste is made consisting of oxide of zinc 100 lb., water 15 gallons, which is saturated with acetic acid of 75 to 80 per cent. strength. The whole is then heated in a pan to boiling and concentrated to 32° to 33° B. (57° to 60° Tw.), sp. gr. 1·285 to 1·300. It is then allowed to settle and the clear solution set aside to crystallise. After some time the crystals, in the form of white plates, are centrifuged, to separate them from the mother liquor, and set to dry on sieves.

Chromium Acetate.—This salt is prepared in solution by mixing solutions of acetate of lead and chromium sulphate or chrome alum ; in the latter case the solution, naturally, contains sulphate of potash.

A solution so prepared is not decomposed on boiling whatever may be its dilution. Whilst cold there is no precipitation by caustic alkalis, alkaline carbonates, phosphates, nor silicates, but on boiling precipitation is complete. How these properties are utilised in dyeing will be seen later on.

Acetate of Copper (*Cupric Acetate; Verdet, Crystallised Vert-de-gris*).—This salt was prepared for a long time by dissolving the basic acetate of copper in acetic acid by the aid of heat, the operation being conducted in a copper pan. After settling, the liquors were decanted and set aside to crystallise, and to facilitate the formation and deposition of crystals, rings of split wood were introduced into the liquid which became covered with crystals. At the present day sulphate of copper is taken as the starting-point. It is converted into acetate by means of acetate of soda, or again it may be prepared from sulphate of copper, the copper of which is precipitated as hydrated oxide and dissolved in acetic acid. In this latter process, milk of lime, in sufficient quantity to precipitate all the copper as oxide, is added to a solution of sulphate of copper in water, taking care to stir the mass well during the addition of the milk of lime; acetic acid is afterwards added, which redissolves the hydrated oxide of copper formed, then the sulphate of lime is allowed to settle and the liquid concentrated until it begins to skin, when it is set aside to crystallise. Another and more rapid method of manufacture, which is preferred to the last process, and by which large crystals of acetate of copper can be obtained, consists in mixing solutions of sulphate of copper and acetate of soda at an average temperature of 70° to 75° C. (158° to 167° F.). In a copper pan, heated by a steam jacket or by a steam coil, containing 800 litres (say 176 gallons) of water, or mother liquors from a previous operation, which are thus saturated with acetate of copper or sulphate of copper, 300 kilogrammes (say 660 lb.) of crystallised sulphate of copper, which are gradually added with constant stirring, whilst the temperature is kept at 70° to 75° C. (158° to 167° F.), are dissolved. In a second vessel made of wood or cast-iron, likewise fitted with a copper coil or a lead coil, 700 litres (say 154 gallons) of water or mother liquors from a previous operation are heated, in which 330 kilogrammes (660 lb.) of acetate of soda are dissolved. The hot solutions are run into a wooden vat, taking care to let the two jets of liquid reach the bottom of the vat at a certain distance from each other, and as gently as possible, so as to avoid setting the mass in motion. The running of the soda solution is regulated in such a way that the salt for equal molecules is always in excess over the sulphate of copper. The affinity of the acetic acid, contained in the acetate of soda, for the copper of the sulphate is so great that the decomposition takes place

somewhat rapidly. After a few hours, according to the season, the temperature of the mixture having lowered to about 34° C. (93·2° F.), the acetate of copper crystallises when the sulphate of soda formed is near its maximum of solubility. The mother liquors are then decanted, and the acetate of copper is then centrifuged. The salt obtained is dried in a stove at a moderate temperature. There is another process which also gives good results, not only as regards the quality of the product obtained, but also in regard to cost of production, manufacturing expenses being lower than by the preceding process. It also consists in utilising the great affinity between the two salts, acetate of soda and sulphate of copper, to form acetate of copper and sulphate of soda, by simply placing crystallised sulphate of copper in contact with a solution of acetate of soda. A solution of 165 kilogrammes (363 lb.) of acetate of soda in 400 litres (88 gallons) of water (or mother liquors from a previous operation) is made in an iron or wooden vessel heated as before by a steam coil to about 80° C. (176° F.). On the other hand, on a double bottom of wood or metallic wire gauze, supported by a frame at a certain height above the bottom of a vat of about 0·4 metre (say 15¾ inches) in depth, of 500 to 600 litres capacity (say 110 to 132 gallons), 150 kilogrammes (330 lb.) of sulphate of copper crystals are laid so that the solution of acetate of soda, once it is run on to them, covers them completely. The beck is then covered, so that its contents remain warm for twelve hours, the time required for the complete transformation of the sulphate of copper into acetate. When the temperature of the liquid has lowered to 34° C. (93·2° F.) the mother liquors are decanted and the acetate of copper, a portion of which has crystallised in fine crystals in the bottom of the vat, is collected, whilst the other portion remains on the double bottom. The acetate of copper formed is centrifuged and washed in the basket of the centrifugal with lukewarm water, then it is dried at a gentle heat for twenty-four to thirty-six hours and afterwards sifted, according to the requirements of customers. The lumps which remain on the sieve are crushed and yield a duller and more bluish acetate of copper. In the two processes just described, the two mother liquors are set to crystallise afresh for the sulphate of soda to crystallise out. After four to five days this salt is separated from the mother liquor which is then again utilised in the manufacture of acetate of copper. The crystallised sulphate of soda is redissolved, to be purified, in a pan, where it is heated with iron turnings so as to precipitate the copper, which soils the sulphate of soda; a small amount of carbonate of soda is then added to precipitate the iron. After twenty-four hours the solution is decanted at a temperature of 30° to 35° C. (86° to 95° F.), and, as the quantity of

water (or mother liquors) from the sulphate of soda is calculated so as to have a saturated solution at that temperature, the sulphate of soda is ready to crystallise; it is then made to undergo a sort of crystallisation in motion (so as to obtain small crystals which are, so to speak, chemically pure). The copper mixed with the oxide of iron is collected and is utilised in the manufacture of sulphate of copper.

Verdigris or Dibasic Acetate of Copper.—This salt is prepared on a commercial scale in the south of France, especially in the neighbourhood of Montpellier, by oxidising in the air plates of copper laid in the centre of the marc from grapes.

Neutral Acetate of Lead or Salt of Saturn (Sugar of Lead).—This salt is obtained by dissolving litharge in acetic acid by direct oxidation of lead by the air in contact with acetic acid. The acetic acid used in both cases is commercial 40 per cent. acid. (1) *By litharge.*—The preparation of acetate of lead, by means of litharge, consists in bringing to about 50° C. (122° F.) a certain quantity of acetic acid run into a wooden or iron vat lined with lead [heating being effected by simple bubbling of the steam in the liquid (that is by a perforated steam pipe)], then by gradually adding litharge (with constant stirring), the weight of which corresponds to five or six-tenths that of the acid; when neutralisation is complete, which is determined by litmus paper, it is allowed to settle, after which it is decanted into a copper pan fitted with a steam coil. The clear solution is then concentrated to 45° to 48° B. (90° to 100° Tw.), sp. gr. 1·45 to 1·500, and it is allowed to settle for a few moments, then the clear liquid is drawn off and set aside to crystallise in earthenware tureens. After twenty-four hours' cooling crystallisation is complete. The tureens are reversed to drain off the mother liquors and the crystals centrifuged and laid aside to dry on a frame in a stove with a strong draught of air, but heated to a temperature not exceeding 35° C. (95° F.), so as not to liberate a portion of the acetic acid contained in the neutral acetate, a salt which is not stable at a higher temperature. Certain manufacturers have modified this process by utilising the property which litharge possesses of being much more soluble in acetate of lead than in acetic acid. The basic acetate of lead so formed is transformed into neutral acetate by adding acetic acid until the solution is neutral. Operations are commenced by running into the solution tank a third of its capacity of acetic acid of 30 per cent. strength, which is heated until it begins to boil; then by means of a sieve, litharge, to which enough water has been added to form a paste, is introduced whilst stirring constantly and in quantity to saturate double the amount of acetic acid run into the tank. That done, the liquid is brought to the boil for a few minutes by a perfor-

ated steam pipe bubbling into the liquid. The litharge dissolves rapidly and completely to form a milky solution of basic acetate, which is converted into normal acetate by adding an amount of acetic acid equal to that originally used, whilst still continuing to stir for some time longer. In this way the loss in acetic acid will be insignificant. It is known that the basic acetate of lead is transformed into the normal acetate by Pfunott's reaction, which consists in adding to a test sample of the lead solution a few drops of a 5 per cent. solution of mercuric chloride until there is no precipitate. Acetic acid is therefore added until this point is reached. Finally, the clarified liquid is evaporated in lead-lined or tinned vessels up to 50° B. (106° Tw.), sp. gr. 1·53, boiling. It is set aside to crystallise in earthenware crystallisers or in shallow wooden becks. So as to remove the copper which exists as oxide in the litharge, and which dissolves at the same time as the latter, sheets of lead are suspended in the concentration vats to precipitate the copper. (2) *By metallic lead.*—In this process the lead is reduced to a very fine state of division; for that purpose it is first melted in a cast-iron pot, then granulated by causing it to fall into a small tub containing cold water. The lead solidifies, assuming a spongy form. This lead is placed in a wooden column with a perforated double bottom also of wood. This vat, of about 4 metres (say 13·12 feet) high, is slightly conical, its lower and widest diameter being, say, 2 metres (6½ feet). Acetic acid, mixed with a certain amount of mother liquors from a previous operation, is run on to the lead. The liquid reaches the upper perforated bottom placed a few centimetres lower down than the top of the vat; it is thus spread equally over the whole surface of the lead. So as to bring to the lead the oxygen necessary for its transformation into acetate, a current of air from draught of chimney branching in its upper part circulates in the vat between the two bottoms; the oxidation of the lead is accompanied by a disengagement of heat, which facilitates the reaction, started in the beginning, by heating the lead by a jet of steam injected into the vat. The liquors are repassed over the lead until they are neutralised and mark 48° to 52° B. (100° to 113° Tw.), sp. gr. 1·5 to 1·565, according to the season, the solution being at a temperature of 60° C. (176° F.); when it has attained the desired degree of concentration it is slightly acidulated in the vat by the addition of a little acetic acid. After some hours' standing the liquid is decanted and set to crystallise in earthenware tureens, or in rectangular shallow wooden becks, so as to cool the liquid somewhat rapidly and crystallise it in forty-eight hours.

TABLE XI.—SHOWING DENSITY OF SOLUTIONS OF ACETATE OF LEAD OF VARIOUS STRENGTHS AT 15° C. (59° F.)

Density.	°B.	Per cent.	Density.	°B.	Per cent.	Density.	°B.	Per cent.	Density.	°B.	Per cent.
1·0127	2	2	1·1084	14	16	1·2211	26·1	30	1·3588	37·9	44
1·0255	3·5	4	1·1234	16	18	1·2395	27·8	32	1·3810	40	46
1·0336	4·5	6	1·1384	17·5	20	1·2578	29·5	34	1·4043	41·5	48
1·0520	7	8	1·1544	19·2	22	1·2768	31·2	36	1·4271	43·2	50
1·0654	8·7	10	1·1704	20·9	24	1·2966	32·9	38	1·4494	44·7	52
1·0796	10·5	12	1·1869	22·6	26	1·3163	34·7	40	1·4735	46·3	54
1·0939	12·0	14	1·2040	24·4	28	1·3376	36·3	42	1·4968	48·0	56

The crystals are then freed from the mother liquor by draining, after which they are passed to the centrifugal and then to the drying stove. The deposits which are formed in this process contain much carbonate of lead, arising from the carbonic acid in the air. They may be utilised by redissolving them by the aid of heat in acetic acid, which is to be used in a subsequent operation. In the installation of an acetate of lead factory by this process, it is necessary to avoid the use of metals in the construction of the apparatus, *especially iron,* which, being very easily attacked, would introduce impurities in the final product, which would be a great drawback, especially for acetate of lead intended to be used in colour manufacture. According to the quality of the acetic acid used in this process more or less well-defined crystals are obtained; thus, whilst with acetic acid *bon goût* the acetate of lead is obtained in large, well-defined crystals, with commercial acetic acid containing propionic acid, butyric acid, etc., the crystallisation is badly defined, like a cauliflower, owing to the presence of the higher homologues of acetic acid.

Basic Acetate of Lead.—This salt is generally met with in solution under the name of *Extract of Saturn,* or in the form of the dried salt. The dried basic acetate of lead is obtained by heating in a steam-jacketed copper pan 50 litres (11 gallons) of water, in which are dissolved 40 kilogrammes (88 lb.) neutral acetate of lead. To this is slowly added 10 kilogrammes (22 lb.) of litharge, and the whole brought to the boil. The evaporation of the liquid is continued until it becomes syrupy and suddenly solidifies; the stirring is continued energetically, still continuing to heat until it is dry to the touch. Extract of Saturn is prepared in the same way, but concentration is stopped when the liquid marks 35° to 36° B. (64° to 66° Tw.), sp. gr. 1·32 to 1·330. It is allowed to settle and the clear liquid is decanted.

Acetine.—The acetine met with in commerce is simply a glyceric ether; it is obtained by heating glycerine and acetic acid, mixed in the

proportion of 53 per cent. glycerine of 28° B. (48° Tw.), sp. gr. 1·24, and 47 per cent. of pure acetic acid, or 55 per cent. of 85 per cent. acid, on the water bath for twelve to fifteen hours in earthenware vessels, similar to the condensers used for mineral acids. During the time the mixture is being heated, care is taken to stir it from time to time, so that the reaction may be complete. It is then cooled for a few hours and the clear portion siphoned off.

Different Industrial Uses of the Products obtained in the Distillation of Wood and their Derivatives.—The examination of the different uses of the products obtained in the distillation of wood industry entering only in a secondary way into the scheme under which this treatise is framed; only the principal commercial uses of the products described above will be given.

Wood Charcoal.—One of the chief industries in which much wood charcoal is used is that of metallurgy; in that industry advantage is

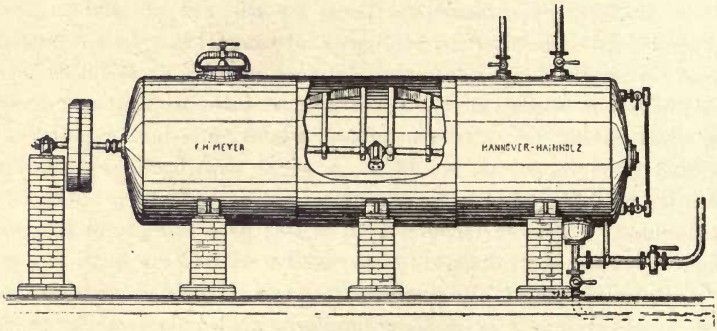

FIG. 51.—Reservoir for mixing wood-spirit.

taken of the properties which carbon possesses of uniting with several metals, such as iron, for the preparation of steel and cast-iron.

Methylic Alcohol or Wood-Spirit.—This is principally used for heating and lighting, and for the preservation of objects; it is used as a solvent for fine oils and certain hydrocarbides, principally resins and gums. Pure methylic alcohol is used for the manufacture of coal-tar colours, like methylaniline, which is obtained by heating wood-spirit and aniline hydrochloride under pressure. Wood-spirit, containing a certain amount of acetone, is used to denature alcohols at a lower excise duty intended for use in industry.

Wood Tar.—The navy and mercantile marine utilise considerable amounts of wood tar for caulking ships, coating ropes, sails and masts. The tar used for caulking is mixed with soft pitch or rosin, and the product so obtained is termed naval pitch or vegetable pitch. Human and veterinary medicine, likewise, use tar in pulmonary and cutaneous affections.

The tar obtained as a secondary product in the preparation of pyroligneous acid is unsuited for the uses just indicated; it is generally fractionally distilled, and yields two or three groups of bodies which are collected apart, for use in the preparation of creosote and carbolic acid, as will be seen farther on.

Acetic Acid.—The monohydrated acid is used in photography and in laboratory work; under the name of *bon goût*, it is used to increase the strength of preserving vinegar, or diluted with water, is used as a substitute for vinegar in cooking operations. The acetic acid of the arts serves as base, as already seen, for the preparation of acetates, aniline, etc. It has a great consumption in tanning and currying for

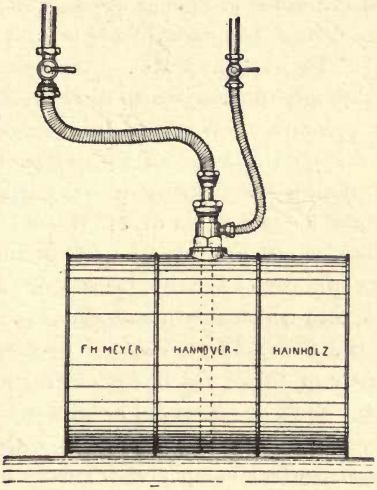

FIG. 52.—Arrangement for withdrawing the products distilled from wood-spirit from iron casks.

the unliming of skins, and in dyeing, as a solvent. In small amount it is used to raise bath for tannin colours.

Acetate of Soda.—This salt is much used in dyeing; it is added to the nitrosamine red development baths, as well as to the printing pastes (tin salt mordants) to preserve the fibres; it is utilised as a reserve for aniline black (Prudhomme's process). For the information of the reader the preparation of a bath for nitrosamine red is given here: 4·160 kilogrammes of nitrosamine red in paste (B.A.S.F.); 2 kilogrammes of hydrochloric acid of 20° B. (32° Tw.), sp. gr. 1·160 (30 per cent. HCl); 50 litres of water as cold as possible; in summer it will even be necessary to add a little ice; 2·800 kilogrammes of sodium acetate and complete to 100 litres with water. Acetate of soda, as a white

reserve for aniline black, is used in the composition of the following bath: 200 of acetate of soda in 150 of water, then make into a paste by heating with 650 of British gum. For a coloured reserve use is made of 500 of a coloured lake of any desired colour, 150 of gum-tragacanth water, in which 150 of acetate of soda is dissolved, and 200 of albumen water 1 in 1. Finally, acetate of soda is used in certain dye baths with acetin.

Acetin.—This product is, at the present time, utilised in large quantities in printing on cotton, because it is a remarkable solvent, whether for basic colours or for colours soluble in alcohol. When steamed, the acetin facilitates the oxidation of the dyes. The following is the composition of what is termed in dyeing *un rongeant*: 350 parts of zinc dust, 350 parts of thickener, 150 parts of acetin, 150 parts of bisulphite of soda of 38° B. (72° Tw.), sp. gr. 1·360. After printing on the tissue, previously passed through the dye bath, it is dried and steamed for ten minutes under pressure, then rinsed, brightened and rinsed once more. Acetin is employed as a salt of tin rongeant in the following proportions: First of all a hot mixture of 550 parts of acetate of tin thickener, 250 parts of acetate of tin of 21° B. (34° Tw.), sp. gr. 1·17, then there are added, whilst cooling, 25 parts of tin salt, 20 parts of acetin and 155 parts of water. Finally, by way of example, the following formula is given, into which acetin and acetate of soda enter, as rongeants, to protect the fibre: 55 per cent. of acetate of tin thickener, 20 per cent. of acetate of tin of 21° B. (34° Tw.), sp. gr. 1·170, 11 per cent. of tin salt, 4 per cent. of acetate of soda, 3 per cent. of acetin and 7 per cent. of water.

Acetate of Ammonia.—This compound is mostly used in medicine or is used to prepare acetamide.

Acetate of Alumina.—Beyond its use in steam colours and in Turkey red dyeing, acetate of alumina is used in waterproofing fabrics. The pieces are passed through swan skin rolls, which moisten them, with a solution of gelatine and acetate of alumina, then on drying rolls; the acetic acid of the acetate disappears and the alumina remains on the fibre.

Acetates of Iron.—These are much used in ink manufacture, the staining of wood black and calico printing. Pyrolignite of iron is the mordant most frequently used in calico printing for dyeing black, violets, grenats, etc.; it is likewise very much used for dyeing silk black or for weighting raw silk. Finally, as the aceto-nitrate of peroxide of iron, it is utilised for dyeing black the silk intended for making the plush or "nap" of hats; this iron compound is prepared by dissolving iron turnings in nitric acid, until a pasty mass of basic nitrate of peroxide of iron is formed. This precipitate is collected and dissolved in acetic

acid by the aid of heat, taking care to preserve a slight excess of precipitate.

Chromium Acetate.—The acetate of chromium in solution is used as a mordant in dyeing cotton. Cellulose possessing the property of attracting sesquioxide of chromium, by a simple immersion of several hours in an alkaline solution of acetate of chromium, a solution is prepared with 100 parts of chromium acetate (of density 1·115, 23° Tw.), and 100 parts of caustic soda (density 1·33, 66° Tw.), and 50 parts of water.

Acetates of Copper.—The acetates of copper are used, as green colours, in oil painting and in dyeing black on wool; they enter likewise into the composition of certain mordants. Acetate of copper is used to make a certain liquor termed *vert d'eau* or *vert préparé*, which is used in the colouring of plans. It enters into the manufacture of stains for wall papers, artificial flowers, etc., and is used in the preparation of Schweinfurt (emerald) green, which is an aceto-arsenite of copper, and is obtained by mixing concentrated boiling solutions of acetate of copper and arsenious acid. Verdigris is used in large quantities in cryptogamic diseases.

Acetate of Lead.—The chief use of acetate of lead is the manufacture of more or less pure chrome yellow, forming the basis of more than thirty lakes derived therefrom. Some of these do not even contain 10 per cent. of lead chromate. It is used in painting and in calico printing. Chrome yellow for painting is obtained by precipitating a solution of acetate of lead by an alkaline chromate. It is then met with as a powder of a fine yellow colour, the shade of which may vary according to the condition in which the precipitation is made. For dyeing fabrics a bath of plumbite of lime is made, by adding 15 to 25 kilogrammes (33 to 55 lb.) of acetate of lead to a milk of lime containing 20 to 30 kilogrammes (44 to 66 lb.) of lime and 500 litres (110 gallons) of water.

Basic acetate of lead is used for loading white silk. The great defect of colours with a lead base is that they blacken when exposed to the action of sulphuretted hydrogen.[1]

[1] And are poisonous.—D. G.

CHAPTER IV

SECONDARY PRODUCTS OF WOOD DISTILLATION

Industrial Derivatives of Wood : Chloroform.—Chloroform, which at the present day is, owing to its anæsthetic properties, a very important product, is prepared by the action of chlorine on wood-spirit in presence of alkalis. The operation is conducted in a still of a capacity triple that of the volume of the liquid to be employed, on account of the secondary reactions that occur, which cause the mass to swell up and froth. From 35 to 40 parts of water at 40° C. (say 3½ to 4 gallons), in which 2 parts (say 2 lb.) of quicklime, previously slaked, and 8 parts (say 8 lb.) of chloride of lime are beaten up, are heated on the water bath. There are afterwards added 1½ parts (1½ lb.) of wood-spirit of 85 per cent. strength, and the temperature of the apparatus is increased rapidly until the still head is well heated. Gas is given off which emulsifies the mass and causes it to froth. The heat is then withdrawn, either by taking the fire from underneath the water bath, or by turning off the steam if heat be applied by a steam coil, then the distillation is allowed to go on of its own accord. Towards the end of the operation heat is again applied to terminate the reaction. The reaction is complete when the liquid which distils has no longer the sweet taste of chloroform.

The distilled product is a mixture of chloroform, methyl alcohol and water ; it forms two layers in the receiver in which it is collected ; the lower layer is chloroform, coloured slightly yellow by the chlorine which it holds in solution ; the chloroform is decanted, washed with water to remove alcohol and then by carbonate of potash. It is finally dehydrated with chloride of calcium, then rectified over concentrated sulphuric acid, which is without action on chloroform, so as to remove the disagreeable odour due to a small quantity of a peculiar chlorinated compound. The water which floats on the chloroform in the condensation of the distilled products may be used again in the preparation of fresh chloroform. Chloroform is more often made from ethyl alcohol, and Laroque and Husant found that, if from 41½ parts of alcohol 550 grammes of chloroform be obtained by the first operation,

640 grammes of that substance will be got in the second operation by using the water distilled in the previous operation, 700 grammes in the third, and finally 730 grammes in the fourth operation. Again, chloroform may be made by distilling acetate of potash and acetone in presence of chloride of lime. Chloroform is a very mobile liquid, colourless, with a mild ethereal odour when it is pure, and a slightly saccharine taste; its density is 1·48. It boils at 60·8° C. (141·4° F.), and burns with difficulty with a fuliginous, reddish flame bordered with green; scarcely soluble in water, though it dissolves in sufficient quantity to impart a most pleasant saccharine taste to the liquid. It is very soluble in alcohol and in ether; its alcoholic solution, run into water, renders the latter milky, whilst pure chloroform gives no turbidity with water. Moreover, pure chloroform should give no turbidity with nitrate of silver, nor coagulate albumen; when it has undergone decomposition, it inflames the skin and becomes poisonous, if it contains chlorine or hydrocarbon oils, arising from bad rectification.

Methyl Nitrate (CH_3NO_3).—This ether of methyl alcohol is extensively used in the manufacture of methylaniline. It is prepared by introducing one part of nitrate of potash into a retort, and running on to it a mixture of two parts of sulphuric acid and one of methyl alcohol; the reaction occurs spontaneously without the aid of heat. A liquid distils which is led into a refrigerating condenser. The oily portion is afterwards washed with water, then rectified on the water bath over a mixture of massicot (PbO) and chloride of calcium, collecting that which passes at 66° C. (150·4° F.). Methyl nitrate is colourless, with a faint ethereal odour; its density is 1·18 (36° Tw.) at 22° C. (71·8° F.). It boils at 66° C. (150·4° F.) and burns with a yellow flame. Very slightly soluble in water, it dissolves, in all proportions, in alcohol and in wood-spirit. Heated to 100° C. (212° F.) with aniline, methyl nitrate is converted into methylaniline-nitrate, which, treated with caustic soda lye, yields methylaniline, which is distilled so as to render it fit for the manufacture of methyl-rosaniline colours.

Ethyl Acetate (*Acetic Ether*) ($C_2H_5C_2H_3O_2$).—Ethyl acetate is met with, already formed, in wine or in wine vinegar. It is utilised in medicine, but is used more particularly to aromatise the vinegars made from acetic acid produced by distillation. It is prepared by the action of acetic acid on alcohol, but, as ètherification by an organic acid is accomplished slowly and incompletely, it is preferred to manufacture ethyl acetate by the intervention of mineral acid, like sulphuric acid, which is made to react on acetate of soda and alcohol. Small quantities of sulphuric acid and alcohol, previously mixed, are run into a retort heated by steam and containing crystallised sodium acetate,

heat is then applied; the liquid distils, and, after passing through a condenser, is collected in a receiver. The distilled product is then agitated with twice its volume of water rendered slightly alkaline, it is then allowed to settle, and the ethyl acetate floating on the top decanted and digested over calcium chloride before being rectified. The chloride of calcium being soluble in this acetic ether, bumping occurs in the still, to such an extent as to render the operation a difficult one. It is therefore preferable, as indicated by Berthelot, to wash the crude product with a weak alkaline solution, then, after rectification, to agitate it with a saturated solution of common salt, dry over carbonate of potash, and finally rectify afresh. The wash waters, containing dissolved acetic ether and alcohol, which have escaped the reaction, are fractionally distilled; the liquid, containing the acetic ether and the alcohol, is collected and used in a fresh operation. Ethyl acetate, the boiling point of which is 74° C. (165·2° F.), is a colourless, very mobile liquid, lighter than water; it has a very pleasant ethereal odour, is slightly soluble in water, and very soluble in alcohol and ether. When it is perfectly dry it may be preserved indefinitely without decomposition, whilst, when it is moist, it gradually decomposes, regenerating alcohol and acetic acid.

Amyl Acetate ($C_5H_{11}C_2H_3O_2$).—The preparation of this ether is carried out in the same way as that of ethyl acetate from acetate of soda, sulphuric acid and amyl alcohol. The rectified product is washed with water, then set aside to dry over calcium chloride and rectified with a little lead oxide. Berthelot advises that the crude product be washed with acetic acid, diluted with its own weight of water, so as to remove traces of amylic alcohol which separate with difficulty; it is then washed with water, dried over chloride of calcium and finally rectified over lead oxide. Amyl acetate is a colourless liquid, boiling at 125°; its density is 0·876; it is insoluble in water, but soluble in alcohol and ether. Its aromatic ethereal odour, recalling the pear, has caused confectioners to use it in certain saccharine preparations. It likewise enters into the composition of some perfumes, so as to impart to them a more pleasant odour, as well as into certain extra fine petroleum products (*pétroles de luxe*).

Acetone (CH_3OCH_3). *Acetone by the Dry Distillation of Acetate of Lime.*—The acetone industry, owing to the numerous applications of this product, is at the present day of some importance. Not only is an appreciable amount consumed in France for the manufacture of *Méthylène type Régie* (that is a mixture of 25 per cent. of acetone and 75 per cent. of methyl alcohol for the denaturation of alcohol for French industrial purposes), but it is also used in the manufacture of celluloid and various

varnishes. Great Britain, likewise, uses an important amount for the manufacture of its smokeless powders ; finally it is used in the manufacture of chloroform. Acetone is produced in the dry distillation of acetates, and especially, as demonstrated by Chenevix, of acetates, the oxides of which can only be reduced with difficulty. The acetates of lime and baryta are those which yield the largest proportion. By heating acetate of lime to a certain temperature it splits up into acetone and carbonate of lime, $(C_2H_3O_2)^2Ca = CaCO_3 + CH_3COCH_3$; but alongside this principal reaction, secondary products are formed from the decomposition of lime salts of fatty acids, homologous with acetic acid, existing in the acetate of lime together with hydrocarbides, due to the presence of tars which, when the temperature goes beyond 400° C. (752° F.), decompose and distil. Moreover, as the acetate of lime often contains an excess of lime, the latter reacts on the acetate to form methane (marsh gas), $(CH_3CO_2)^2Ca + Ca(OH)^2 = 2CaCO_3 + 2CH_4$. Hence a portion of the acetone, which from the amount of acetic acid in the pyrolignite of lime ought to be obtained, is lost, a loss which is increased by the decomposition of a portion of the acetone on the metallic sides of the still. Care, therefore, should be taken in this manufacture to secure a uniform heat and regular but powerful agitation of the contents of the still. Theoretically, 100 lb. of pyrolignite of lime containing 80 per cent. of acetate of lime, ought to yield 30 lb. of acetone ; practically, with the best plant only 25 lb. are obtained, which, after rectification, only yield 20 lb. of acetone per 100 lb. of acetate of lime.

Installation of an Acetone Factory.—As in the case of every industry in which condensers consisting of coils immersed in water are used, it is necessary to build the factory in proximity to as abundant a supply of water as possible. The reservoirs (storage tanks), for the different distilled and rectified products, are installed so as to avoid fire. The stills, boilers, engines, dynamos, pumps are arranged as in the plan of a wood charcoal factory. As just said, the manufacture of acetone consists in distilling acetate of lime in a retort at a temperature which reaches a dull red heat. Water is added to the crude acetone, collected at the condenser, to separate the oils which float to the top, it is then rectified, which yields pure acetone, methylethyl acetone and other acetone oils. The decomposition of acetate of lime is effected in cast-iron retorts fitted with an agitator, as in the manufacture of acetic acid by sulphuric acid (Fig. 53). About 300 kilogrammes (660 lb.) are distilled in one charge in these retorts. The heating of the retort, which some years ago was done in a bath of molten lead, so as to secure a uniform heat, but had the defect of rapidly converting the lead into oxide of lead in contact with the air, is now done by naked fire. So as to avoid overheating and

so as always to secure uniform heating, the hearth, with a fire bridge, is placed at a certain distance from the bottom of the pan, thus forming a rather large heating chamber, to avoid too great variations of temperature by irregular stoking. A dust chamber should be installed between the pan and the condenser to stop the dust produced, which might otherwise obstruct the condenser pipe. For a daily production of 1 ton of acetone, five retorts are required. Above the retorts is a floor on which the pyrolignite of lime is stored, which is afterwards fed into the retorts through a wooden hopper with a channel which enters into the manhole of the retort. In small installations, the retorts are charged by emptying the sacks into this manhole. The pan is heated gently to cherry red, then the acetate of lime is run in, the manhole

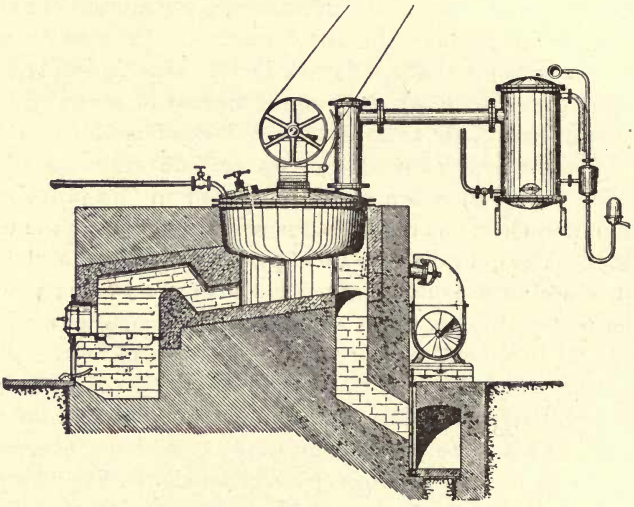

Fig. 53.—Plant for the manufacture of crude acetone.

closed and the lid luted, after which the agitator is put in motion. The first vapours which escape are led to the chimney, because they consist in great part of water; after some minutes a stoppage occurs, which necessitates an increase in the heat, so as to start the distillation properly so called of the acetone. The vapours are then led to the condenser, consisting of rectilinear pipes, connected on the outside with the tank by elbows; this arrangement enables the coil to be easily cleaned, as it always retains entrained dust in spite of the dust chamber in front of it. The condensed liquid has a more or less brown colour, due to the hydrocarbides which it contains and to entrainment of salts of lime in the form of dust. Towards the end of the distillation, the condensed product flows drop by drop, then the goose-neck begins to cool; when this

occurs steam is injected into the retort so as to carry over a portion of the gas and dilute the remainder, so as to avoid the sudden inflammation of the gas contained in the still very hot retort when it is opened. The residue of the operation which is valueless is in the form of a fine grey powder still possessing an empyreumatic odour. The crude acetone is collected in a cast-iron cylinder fitted with an agitator; it has an average density of 0·930 and contains 30 to 60 per cent. of pure acetone. Water is added until it only contains 30 per cent. of pure acetone, then slaked lime, and the liquid is stirred for some time to neutralise free acid; it is then allowed to settle, when the solution separates into two layers: (1) A lower layer which is a dilute solution of acetone; (2) an upper and darker layer (due to the tar that it contains), which consists principally of acetone oils, the major portion of which is thus separated from the aqueous layer. Each of these liquids is sent to a different storage tank. The acetonic liquid is then distilled in a column still, taking care to separate the first runnings, which are slightly coloured and contain aldehydes, amines, etc., from the milky last runnings, which contain the higher acetones. There are collected apart: (1) The products which after the first runnings still contain oils and become turbid on addition of water; (2) those which mix in all proportions with water, yet decolorise permanganate in solution; and finally (3) the most important as regards quality, products which give no reaction with permanganate of potash and which are put on the market directly. The portions which contain the oils are agitated with water once more so as to separate them; the solution containing a little acetone is run into the crude acetone store tanks to be re-treated. To purify the fraction decolorising permanganate, the amount of this salt necessary for treating a given volume is determined in the cold state, then the corresponding weight of permanganate solution is added to the acetone, which is again rectified. In this distillation, which ought to be carried out with care, the portions of pure acetone, which pass between 56° and 58° C. (132·8° to 136·4° F.), are collected apart for use in the manufacture of chloroform and iodoform. The acetone thus prepared marks 98 to 99 per cent.

Treatment of the Tar. Distillation of the Crude Creosote.—The tar, separated from the crude pyroligneous acid by decantation, as well as that precipitated in the distillation of pyroligneous acid, forms a mixture consisting of water, saturated hydrocarbides, whether liquids, or solids like paraffin, compounds of the aromatic series, like benzol, toluol, carbolic and cresylic phenols as well as naphthalene. When it is simply sought to free the tar from water and from light products, so as to put it on the market, a copper pan, heated by a coil (Fig. 54), is

used, in which the distillation is conducted with precautions to avoid priming of the tar. The water distils over, bringing acetic acid, alcohol and light oils in its train, until the distillate flows drop by drop. From 20 to 25 per cent. of distillate is collected in this way. The tar is afterwards run off from the apparatus, passing it through a cooler, before storing it in barrels. According to whether the tar comes from the partial carbonisation of wood, or from distillation in closed vessels, it possesses different properties, which properties, again, vary with the nature of the wood distilled. When it is desired to distil the tar completely, so as to extract the phenolic oils (or crude creosote) from it and the pitch, the crude tar is distilled in cast-iron stills similar to those used for petroleum residues, but smaller, 39 inches wide by 78 inches deep

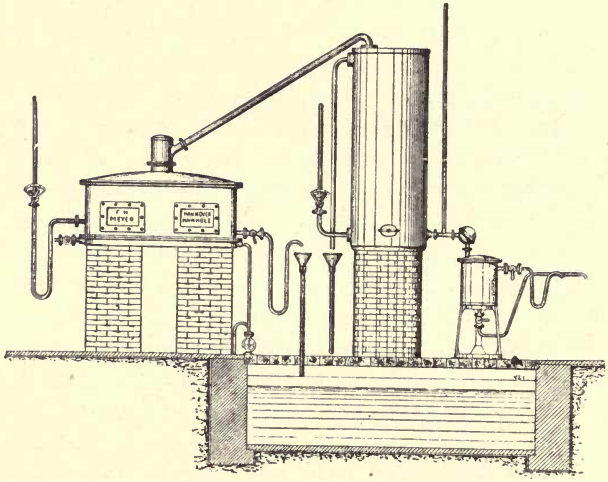

Fig. 54.—Continuous working plant heated by steam for dehydrating wood tar.

(Fig. 55). These stills are cylindrical, with a concave bottom ; they have a pipe in the bottom, with a tap for running out the pitch, protected from the action of the fire by a small masonry arch, and wide enough to obviate all hitches in running out the pitch. Wrought-iron taps suit very well for this class of work. The lid, surmounted by a dome, the goose-neck, and all which follows it, are generally made of copper. At the end of the condenser is a siphon, which acts as a liquid seal, to allow the uncondensable gases to escape to the outside, or to be aspirated by a pump and propelled to a gasometer, from which they issue through special piping, to be burned in the furnaces. The still being charged with about 1 ton of tar, the mass is heated rapidly until the copper still-head commences to become lukewarm, the fire is then moderated, and the distiller, to avoid all priming, waits until the first

portions distilled issue from the exit of the condenser. The distilled products are collected in a separator (Fig. 56), a sort of cast-iron case

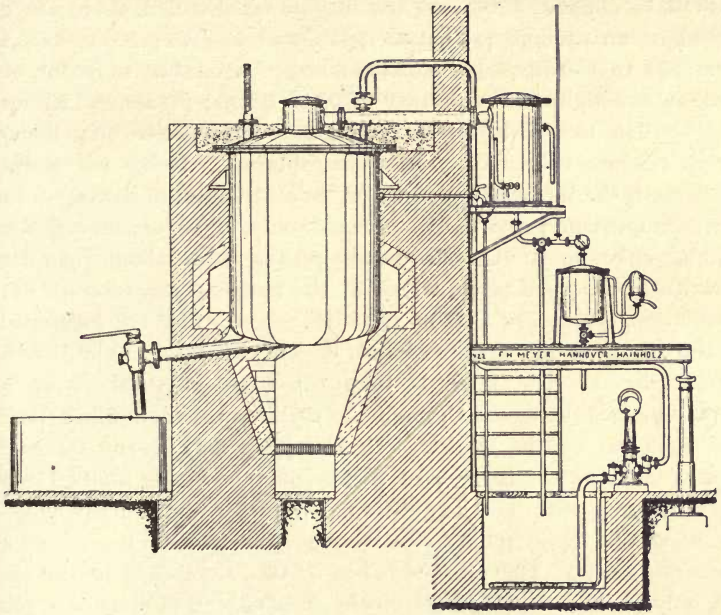

FIG. 55.—Cast-iron retort for the distillation of tar.

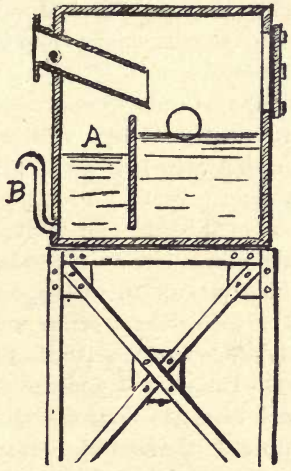

FIG. 56.—Wood-tar separator.

containing a vertical partition, which forces the aqueous portion to separate from the oils. All these products are fractionated into different categories, according to whether they contain both alcohol and acetic

acid, acetic acid alone, and, finally, crude creosote. The distillation lasts twelve to fifteen hours according to the products which it is desired to obtain. From the quantity of tar distilled, there are collected on an average (*a*) between 110° and 120° C. (230° to 248° F.), from 100 to 150 litres (22 to 33 gallons) of a mixture of water, acid, alcohol, and light oils of density 0·700 to 0·800; (*b*) above 120° up to 230° C. (248° to 446° F.), from 200 to 300 litres (44 to 66 gallons) of heavy oils are collected. These oils contain 10 to 15 per cent. of creosote, if the tar be the product of the distillation of hardwood (oak, hornbeam, beech); and 6 to 7 per cent. only, in the case of soft woods (birch, poplar, chestnut). The residue in the retort, about 6 per cent., constitutes the wood pitch, which at the temperature at the end of the distillation, 260° to 280° C. (500° to 536° F.), is very fluid but inflammable in the air; it is allowed to cool for several hours in the still before discharging it. The pitch is then run into extinguishers, or into petroleum barrels, sawed through the middle, which are filled directly and thus put on the market. Or the pitch may be run into a flat cooling tank, which, to prevent the product becoming kindled in the air, is covered with a wrought-iron plate, with only an opening for receiving the liquid pitch; after cooling, the pitch is broken up and packed in casks. The pitch obtained by the distillation of the tar is not only used in making coal blocks (briquettes), like coal-tar pitch, but it is also employed in the manufacture of artificial asphalt, certain varnishes, and coatings, like the caulking used in the mercantile marine or navy. As to the heavy tar oils, chiefly from beech tar, they are the source of creosote and guaiacol.

Purification of Creosote.—Crude creosote is again distilled, to separate first the 30 per cent. of acetic acid which it still contains, then the portions passing between 195° and 220° C. (383° to 428° F.), which constitute the phenic (carbolic) acid, the cresylic phenol and the creosol. The oil obtained being somewhat coloured, it is treated with its own volume of a solution of caustic soda of 36° B. (53° Tw.), sp. gr. 1·265, in a washing apparatus, with a conical bottom, bearing in the interior (Fig. 57) a helical agitator for mixing the mass and a steam coil to heat the liquid, so as to separate the greater part of the heavy oils in suspension. The phenol and the creosote combine with the soda. It is allowed to settle for some time, then the aqueous solution is decanted into a still, into which steam is injected to expel the hydrocarbides which have not combined with the soda. Steam is injected into the still until a sample, taken from the interior, is limpid like water and does not turn milky on the fresh addition of water. The solution is allowed to cool, and the oily portion which

floats to the top is again decanted, then the soda solution is treated by a mineral acid, like hydrochloric acid or sulphuric acid, or, better still, by carbonic acid, which, in this case, is passed in at the lower part of a column, whilst the soda solution is run in at the top. The creosote liberated in this way is fractionally distilled in copper stills with column rectifiers (Fig. 58), so as to obtain a first separation of the phenic (carbolic) acid, collecting apart that which distils between 150° and 195° C. (302° to 383° F.), then the liquid passing between 195° and 220° C. (383° to 428° F.) which is chiefly creosote. This latter portion undergoes three different rectifications in the column apparatus. In each rectification the products passing between the same temperatures

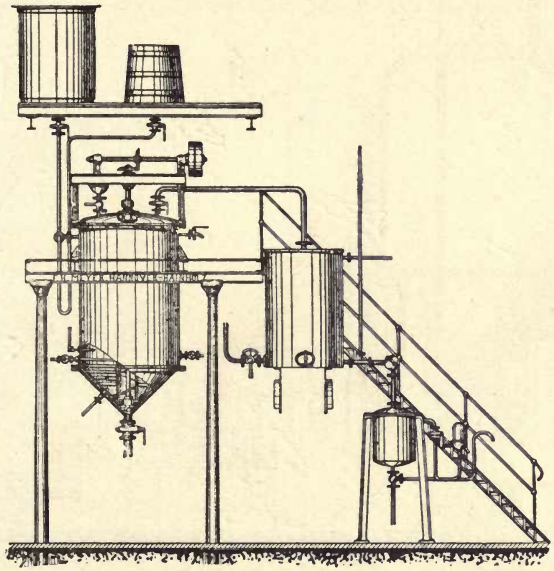

FIG. 57.—Agitator for the treatment and steam distillation of wood-tar oils.

are collected in the same reservoirs : (*a*) 195° to 205° C. (383° to 401° F.); (*b*) 205° to 215° C. (401° to 419° F.) ; (*c*) 215° to 225° C. (419° to 437° F.). This necessitates three reservoirs for the products of the first rectification, three others for those of the second rectification, and, finally, three other reservoirs for those of the third rectification ; into these are run the products passing between the same temperatures and coming from the rectification of one of the three liquids collected in the previous rectification. Finally, three fairly homogeneous liquids are obtained : (*a*) the products boiling between 195° and 205° C. (383° to 401° F.) or 20 per cent. guaiacol ; (*b*) the products collected between 205° and 215° C. (401° to 419° F.) which constitute crude officinal 80 per cent.

creosote ; (*c*) between 215° and 225° C. (419° to 437° F.) the creosote from the last runnings. These products, after a fresh combination with soda of 36° B. (66° Tw.), sp. gr. 1·33, then a distillation by entrainment with steam, as already described, and finally decomposition with 50 per cent. sulphuric acid, avoiding a heating of the mass, result in the preparation of prime quality compounds. To obtain white creosote, the creosote previously obtained is distilled *in vacuo ;* a yellow creosote is obtained, which is rectified a second or a third time if need be. To prepare crystallised guaiacol the 20 per cent. guaiacol is distilled. The

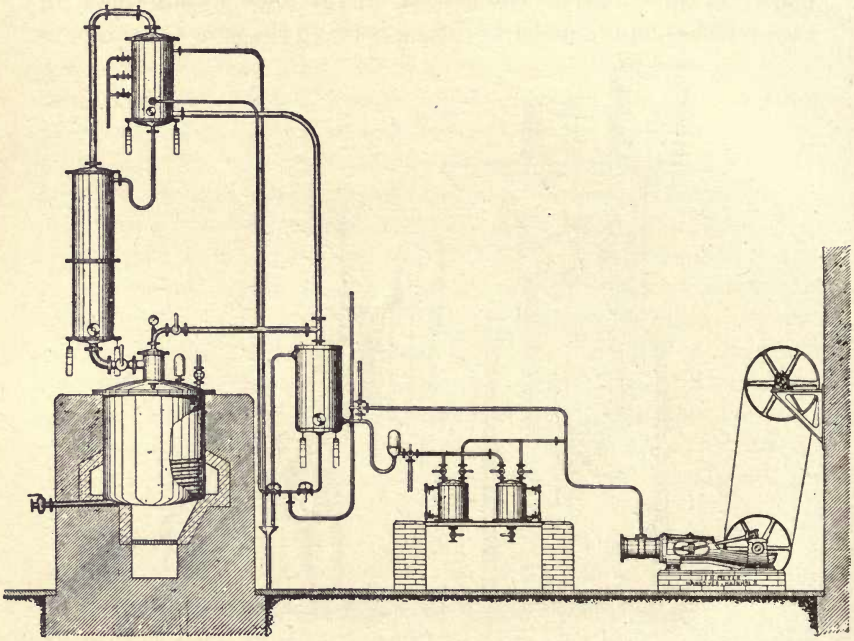

Fig. 58.—Still with column for fractionating tar oils heated by naked fire or by steam at the atmospheric pressure or *in vacuo.*

monophenols are first separated, then the liquid collected afterwards is placed in glass vessels, in a freezing machine, where it is refrigerated. The crystals so obtained are centrifuged in a small enamelled turbine.

Utilisation of Wood Waste and of Waste from Wood Charcoal Factories. Utilisation of Wood Sawdust.—Wood sawdust, which is a cumbrous waste product in certain industries, where it cannot be directly used as fuel, although at the present time ingenious furnaces with large grate surface arranged in stages are installed, may be profitably utilised in many ways. When a sufficient quantity is collected to warrant the erection of a first installation, it is distilled, as already

described, so as to extract wood-spirit, acetic acid and powdered charcoal. The manufacture of oxalic acid, according to Capitaine and Herlings' process, and of glucose, then of ethyl alcohol, consume a certain quantity. Again, wood sawdust is used in the purification of gas and in the filtering of oils purified by sulphuric acid. Finally, excellent coal blocks or briquettes are made from it, although it is difficult to get it to segregate, by mixing it with coal tar, glue and an alkaline rosinate. In Austria, wood sawdust is dry heated until the moment before it begins to gasefy, when the tarry ingredients, which it contains, separate from it; they are then ready to serve as an agglutinant. The sawdust is at once passed to the hydraulic press which moulds it into briquettes, sufficiently consistent to bear transport without crumbling.

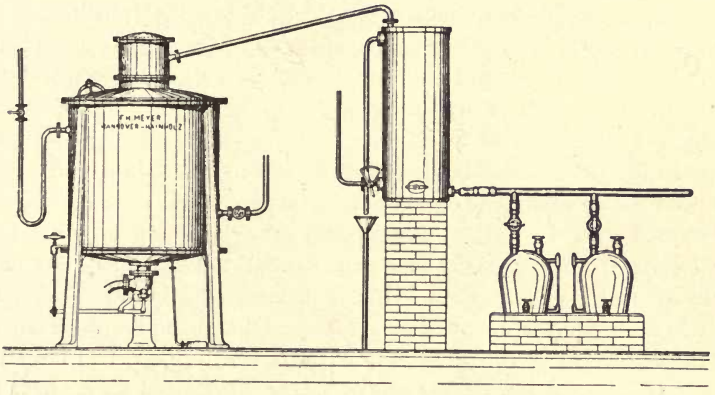

Fig. 59.—Plant for the distillation of creosote.

Manufacture of Oxalic Acid.—Oxalic acid is produced in numerous reactions and, more especially, by the oxidation of organic matters. The action of nitric acid on alcohol, glycol, sugar, starch and on cellulose yields oxalic acid. In the same way fused potash acting on sugar, starch, pectic acid and on wood sawdust produces oxalic acid. It is this latter reaction, indicated by Vauquelin, then by Gay-Lussac, that is utilised commercially, at the present time, to produce oxalic acid very cheaply after the old process of Roberts, Dale & Co., of Manchester, on which some improvements have been made. The wood sawdust, from which oxalic acid is made, is first brought to the state of paste by mixing it with an alkaline solution marking 37° to 38° B. (69° to 72° Tw.), sp. gr. 1·345 to 1·360, and consisting of potash and soda in the proportion of one equivalent of the former to two equivalents of the latter. When

it is desired to make oxalate of potash only, then caustic potash alone should be used. To 100 lb. of sawdust, 300 to 350 parts of real alkali (R_2O) are added in solution. In the beginning of this industry the paste obtained was spread on thin layers on iron plates, which were gradually heated from underneath, care being taken to stir the mass. Now, however, this carbonisation is effected in a horizontal cylindrical retort, capable of revolving on its axis, in which an archimedean screw revolves in an opposite direction. This retort being heated to dull redness, the mixture swells and intumesces during the evaporation, with generation of gases, which are burnt underneath the furnace. The archimedean screw, which continually shifts the mixture of sawdust and alkali, conveys the carbonised material to the other end of the retort, whence it is run into trucks which convey it to the lixiviation house, where the alkaline oxalate is extracted. For this purpose the charred mass is taken up by a certain amount of water at 10° to 20° C. (50° to 68° F.); this dissolves the alkaline oxalates, which are more soluble in cold than in hot water. The liquid is, after setting, decanted and evaporated to dryness. The oxalates are again taken up by cold water, the solution brought to the boil and treated by lime to precipitate oxalic acid, and regenerate the caustic alkalis which remain in the liquid and can be utilised for a subsequent operation. The precipitate of oxalate of lime is washed with water, then decomposed by a slight excess of sulphuric acid so as to liberate oxalic acid. According to Chandelon, one molecule of oxalic acid requires three molecules of sulphuric acid. The salt is rubbed up with water to a thin paste, and the requisite amount of sulphuric acid at 15° to 20° B. (23° to 32° Tw.), sp. gr. 1·115 to 1·160, is added with stirring. After adding water it is heated for a few hours, then filtered to separate the sulphate of calcium. The solution of oxalic acid, after evaporation, is left to crystallise in lead vessels; but, as the crystals of oxalic acid so obtained are slightly coloured, they are purified by simply redissolving them in water and recrystallising them. The residue, left by exhausting the carbonised mass with cold water, is calcined in a reverberatory furnace; it yields a mixture of carbonate of potash and soda, which is caustified by lime for use in a subsequent operation. From 100 lb. of wood sawdust 50 to 60 lb. of oxalic acid are obtained, and as in this process the alkalis are used over again, the greatest expense is coal, of which 40 lb. are required to make 1 lb. of oxalic acid, which enables oxalic acid to be prepared 50 per cent. cheaper than that obtained by the action of nitric acid on starch or on molasses. Gouley, in his process, subjects the sawdust to a preliminary purification by levigating it with boiling water, which extracts the tannic acid which it contains. The yield, he says, is increased.

TABLE XII.—SHOWING THE AMOUNT OF OXALIC ACID OBTAINED FROM THE SAME QUANTITY OF SAWDUST TREATED BY MIXTURES OF POTASH AND SODA IN DIFFERENT PROPORTIONS

Ratio KHO to NaHO.	°C.	Oxalic Acid per cent.	Ratio KHO to NaHO.	°C.	Oxalic Acid per cent.
0 : 100	200-200	33·14	40 : 60	240-250	80·57
10 : 90	230	58·36	60 : 40	240-250	80·08
20 : 80	240-250	74·66	80 : 20	245	81·24
30 : 70	240-250	76·77	100 : 0	240-250	81·23

Oxalic acid crystallises with two molecules of water of crystallisation. It drys completely, but very slowly, in a dry atmosphere. It is used in very large quantities in calico printing so as to remove, in certain points, either the mordant or the dye, so as to obtain by the absence of colour on the points impregnated with oxalic acid any desired design. Oxalic acid is also used to make blue ink from Prussian blue.

TABLE XIII.—SHOWING DENSITY OF SOLUTIONS OF OXALIC ACID OF DIFFERENT STRENGTHS AT 17° C. (62·6° F.)

Density.	Per cent.	Density.	Per cent.	Density.	Per cent.
1·0035	1	1·0210	6	1·0385	11
1·0070	2	1·0245	7	1·0420	12
1·0105	3	1·0280	8	1·0455	13
1·0140	4	1·0315	9	1·0490	14
1·0175	5	1·0350	10	1·0525	15

Manufacture of Briquettes (Coal Blocks) from Wood Charcoal.—The waste from the carbonisation of wood char dust and the smalls produced in the carbonisation of twigs, have been used for some years in making small briquettes or coal blocks, sometimes termed Paris coal. In making these briquettes, glue or pitch is generally used as an agglutinant, and saltpetre to make them burn more readily. The charcoal waste is first ground and sifted and then triturated with saltpetre and a solution of strong glue (when glue is used) of 15 per cent. strength, in a circular trough by two conical cast-iron grooved millstones. When soft pitch or hard pitch is used to make this block fuel, the char dust, the pitch and the saltpetre are ground in the mill, then the mixture is heated in a mixer by steam jets which penetrate into the mass, melt the pitch and bring the paste to the right consistency for good moulding. This paste is then fed into a distributor (Fig. 60), the dimensions of which are calculated so as only to let enter into the compression

mould the quantity of the mixture necessary to make briquettes from it, which are then passed to the stove. For that purpose they are piled on trucks with shelves, which are run into a continuous drier, arranged so as to dry them gradually. The trucks enter the end of a long masonry alley, which acts as a stove, and leave it by the hottest part. A current of hot air, heated to about 70° C. (158° F.) by branched pipes, circulates in an opposite direction to the trucks. In the manufacture of Paris coal, the charcoal waste, previously moistened with 10 to 12 per cent. of water, is reduced to the state of rough powder; it is then triturated in the trough of a millstone mill, with about 30 per cent. of coal tar or molten soft pitch. Certain factories even use pulverised hard pitch. When the paste appears quite homogeneous, it is

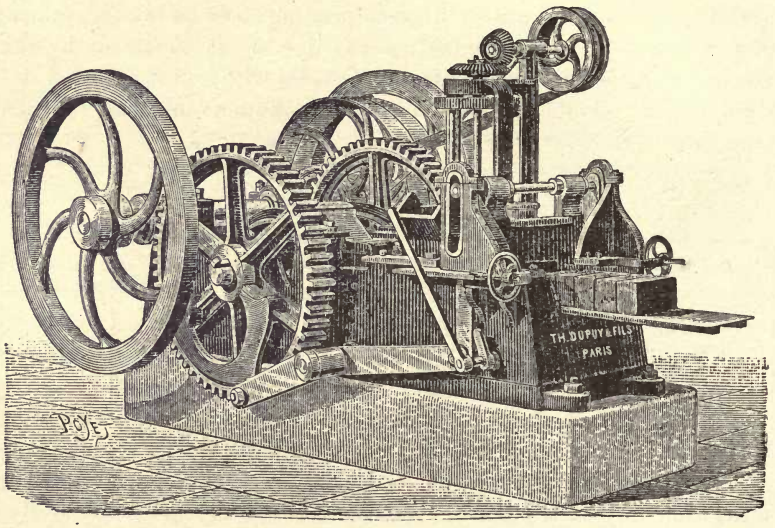

Fig. 60.—Machine for making wood charcoal briquettes or coal blocks with horizontal piston.

shovelled into a special machine (Fig. 61), from which it issues moulded into small cylinders on an endless chain, from which they are taken, to be placed in wrought-iron boxes of a certain sort. The boxes, full of cylinders of moulded paste, are afterwards furnaced in one of the muffles of a furnace, where they are carbonised; during this carbonisation the hydrocarbides of the tar are decomposed, gases are given off, which escape through holes made in the muffle, and are conducted back into the furnace, where they may suffice for the production of the heat necessary to complete the carbonisation of the coal block. This muffle furnace may be continuous. Carbonisation is complete when no more smoke is disengaged from the holes made in the muffle. The contents

of the muffle are run into wrought-iron extinguishers, the lids of which are luted so as to avoid all access of air, which would infallibly lead to a consumption of fuel in sheer loss; six or eight hours afterwards the extinguishing is complete, and the product is sent to the warehouse. Paris coal having the advantage, when a bright quick fire is not desired, of yielding a more easily regulated heat, is much used in laboratory operations and in domestic economy.

Manufacture of Carbonate of Potash (K_2CO_3).—Among the different commercial sources of which advantage is taken to manufacture carbonate of potash there is one, *viz.*, the ashes of plants, which falls within the scope of this treatise. The greyish residue termed ash, left by plants when burnt, contains potassium, generally as carbonate mixed with the chlorides, sulphates, phosphates or silicates of the different alkaline bases. The carbonate of potash is separated from the other salts by systemati-

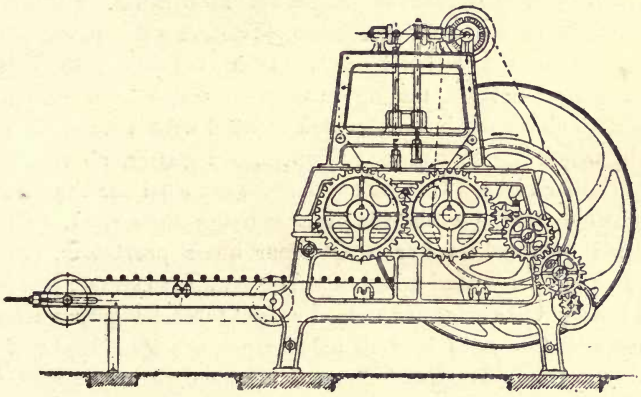

Fig. 61.—Machine for making Paris coal and briquettes for small heating appliances.

cal levigation. All plants are far from leaving ashes of the same composition; those which grow on the sea-shore particularly contain soda salts, whilst inland plants are especially rich in potash. As to the amount of ashes produced, it likewise varies according to the source of the plants and the nature of the soil on which they have grown. In this way herbaceous plants leave a larger residue on ignition than ligneous plants.

Table XIV.—Showing the Average Percentage of Ash in Certain Plants

Plant.	Ash per cent.	Plant.	Ash per cent.	Plant.	Ash per cent.
Alder . .	0·40-0·55	Oak . .	1·80-3·3	Vine twigs .	4·60-7·75
Hornbeam .	0·60-1·30	Charlock .	4·00-4·5	Pea stems .	11·30
Pine . .	0·80-1·10	Wheat Straw .	4·50-6·3	Potato stems .	15·00

The ashes contain a soluble portion consisting of carbonate of potash, sulphate of potash and chloride of potash accompanied by traces of silicates, and an insoluble portion consisting more especially of carbonate of lime with a little phosphate and silica. The incineration of plants, for the extraction of potash, is practised in countries where forests abound and the means of transporting wood difficult, as, for example, in certain American countries. The herbaceous plants which cover the immense steppes of Russia, and the brushwood, which is supplied by the German or by the Vosges forests, are likewise a source of carbonate of potash. The plants, dried by long exposure to air, are burnt, either in pits about 1 metre (3·28 feet) deep, or on plane, well-beaten surfaces sheltered from the wind. Their combustion should be slow, because with too brisk a fire a part of the ash would be scorified (clinker) by the silica contained in the plants. The fire is fed until the pit is full, or until there is on the plane surface a sufficient amount of ashes. These are afterwards screened before being lixiviated. The ashes are lixiviated in casks or in vats, having the form of a reversed truncated cone, fitted with a perforated double bottom, resting on supports which keep it about a few centimetres above the bottom, which is fitted with a bung or a lateral tube, by which the lye can be run out into a gutter, above which are several similar casks. Generally six casks are used for the levigation; one is being charged whilst the other is being discharged. The ashes, moistened with cold water twenty-four hours previously, are lightly packed on the perforated false bottom covered with straw; this addition of cold water to the dry ash not only facilitates levigation afterwards, but increases the yield in carbonate of potash; the silicate of potash is decomposed, under the influence of the carbonic acid of the air, into carbonate of potash and silica.

The ashes, contained in the freshly charged vat, are covered with straw and washed with a feeble lye from a vat, the exhaustion of which is nearly complete. A vertical pipe, rising from the double bottom and passing through the ashes, allows the air to escape and make room for the liquid. The vat is thus filled and started; about four hours afterwards the lye, which then contains 30 per cent. of soluble salts, is run off and evaporated. The exhaustion is stopped with the last vat, which is allowed to drain; its solid residue constitutes *charrée*, which, in virtue of the phosphate of lime and organic matters which it contains, presents a certain value as a manure; it is also used in the manufacture of ordinary bottle glass. In France, in some factories, the old lixiviation process is still pursued; the vats used have neither false bottom nor stopcock, but in the centre of the bottom is a hole in which a central pipe, open at both ends, is fixed, and formed of several trunks, which

can be separated and withdrawn as desired. The ashes, which fill two-thirds of the vat, are intimately mixed with the liquid, which is added thereto for lixiviation. They are then left to rest for some hours. The solid matters are deposited below a layer of clear liquid, which is decanted by removing from the central pipe a sufficient number of pieces; when the operation is finished, the pipe is fixed up again for a second levigation, and so on.

Evaporation of Lyes and Calcination of the Salts.—The lyes are evaporated in shallow, wrought-iron or cast-iron pans, into which fresh lye is being continuously added, until a sample of the liquid solidifies whilst assuming a crystalline form. At this moment the fire is moderated. A brown crystalline crust is deposited on the sides of the pan, which goes on increasing in proportion as the evaporation proceeds; when it is finished, the pan and contents are allowed to cool, and the crude dark brown potash, still containing 6 per cent. of water, is detached with a chisel. It is termed *flux, salin* or *potasse cassée* (crushed potash). This method of evaporation speedily wears out the pans, by the overheating to which they are exposed, when the salt adheres to the bottom (this may also cause an explosion). A better process is to stir the liquid continually with a poker as soon as it assumes a pasty condition. Drying is thus facilitated and any deposit of salt on the sides of the pan is prevented. The potash obtained in this way contains 12 per cent. of hygrometric water and is known in commerce as *potasse brassée.* In certain factories the greater part of the sulphate of potash which accompanies the carbonate of potash in the lyes is separated by taking advantage of the different solubility in the two salts. For this purpose the lyes are concentrated to the desired extent; they are then left to cool in wooden vats which are frequently stirred. The greater portion of the sulphate of potash is deposited in crystals; the supernatant solution is then decanted and evaporated as described above. In certain German factories three pans are used to evaporate the lyes. A start is made by heating the liquid in the first pan, that farthest from the furnace, then it is passed to the second pan, where it is brought to a syrupy consistency. Finally, the evaporation is completed on the sole of a reverberatory furnace.

Calcination of the Crude Potash.—The object of calcination in the air is to destroy all the organic matters which the salt contains and which discolour it, and to eliminate the 6 per cent. of water which it contains. The operation is conducted on the sole of a reverberatory furnace, capable of taking a charge of 1·2 metric tons (say 24 cwt.) of crude potash, heated to dull redness by lateral fires, and fitted with a

draught chimney in front and above the opening of the sole of the reverberatory furnace. A start is made by heating the walls of the furnace, then the crude potash is introduced, generally dividing the charge into tiers, and spreading it uniformly over the whole surface of the sole. The salt is stirred by a crowbar through a loophole, on a level with the sole of the furnace, so as to facilitate the dehydration and promote the access of the air which burns up the organic matter. A slow fire is kept up, stirring briskly so as to prevent the mass fusing. The salt whitens gradually, and when the operation draws to a close, that is to say after about six hours' heating, a workman crushes the lumps by aid of an iron spatula. The granulated potash is then withdrawn by a hoe and is left to cool on a space for the purpose in front of the furnace. Potashes so obtained are sometimes coloured red, yellow, or bluish-green, by oxide of iron or manganese. This shade is generally distinctive of its origin. The finest potash is white. It is termed pearl ash (*perlasses, cendres perlées*).

Refining.—When it is desired to extract commercial carbonate of potash from crude potashes, they are treated in the cold state with their own weight of cold water; the carbonate of potash, which is very soluble, dissolves almost alone, whilst the sulphates, chlorides, etc., remain insoluble, owing to their slight solubility, in a solution of carbonate of potash. The liquid, after decantation and evaporation, yields refined potash or commercial carbonate of potash, which always contains a little carbonate of soda.

CHAPTER V

ANALYSIS OF RAW MATERIALS AND FINISHED PRODUCTS

THE analytical methods used in the industrial chemistry of wood, to be described in this chapter, may be divided into two sections: A. Analysis of Raw Materials; B. Analysis of Finished Products.

A. *Analysis of Raw Materials*: 1. *Lime.*—Lime is used, as already mentioned, to neutralise the pyroligneous distillate; it is also used, in small quantity, in the rectification of wood-spirit. For the first purpose, which is the most important, so as to obtain the best yield, the lime ought to be free from magnesia, since 100 parts of acetic acid only yield 118 parts of acetate of magnesia instead of 131 parts of acetate of lime. Moreover, the lime ought to be almost entirely soluble in acetic acid. Consequently it should contain but little silica and silicates, which, during the preparation of pyrolignites, increase the amount of mud in the filter presses, which one cannot dream of washing on account of the expense entailed. A large percentage of magnesia and silicates is recognised by the way the lime slakes in water; a complete quantitative analysis will determine in a more sure way the amount of impurities present in the lime. Beaumé's areometer dipped into a solution of 100 grammes of quicklime per litre (1 lb. per gallon) will give rapidly and exactly enough the percentage of lime by the following table:—

TABLE XV.—SHOWING THE PERCENTAGE OF (CaO) QUICKLIME IN MILK OF LIME OF DIFFERENT DENSITIES

°B.	Density.	Lime in 1 Litre in Grammes.[1]	°B.	Density.	Lime in 1 Litre in Grammes.[1]
1	1·007	7·5	14	1·108	137
2	1·014	16·5	15	1·116	148
3	1·022	26	16	1·125	159
4	1·029	36	17	1·134	170
5	1·037	46	18	1·142	181
6	1·045	56	19	1·152	193
7	1·052	65	20	1·162	206
8	1·060	75	22	1·180	229
9	1·067	84	24	1·200	255
10	1·075	94	26	1·220	281
11	1·083	104	28	1·241	309
12	1·091	115	30	1·263	339
13	1·100	126			

[1] Or lime in 100 imperial gallons in lb.

Quantitative Analysis: (1) *Water.*—Ten grammes of the pulverised lime are dried at 110° C. and the loss determined.

(2) *Insoluble in Acetic Acid.*—One gramme of the pulverised lime is dissolved in acetic acid, boiled, filtered through a tared filter, dried and weighed.

Total Alkali.—A few drops of litmus or phenol phthalein are added to the milk of lime diluted with water, and normal hydrochloric acid added until complete neutralisation is effected.

Magnesia.—The amount of magnesia present in lime is determined by the difference in the results previously found with the lime, estimated by Mohr's process, which consists in taking 25 c.cm. of milk of lime at 2 grammes per litre, to which 25 c.cm. of decinormal oxalic acid is added. Ammonia is then added drop by drop so as to render the liquid slightly alkaline; it is then boiled to precipitate the lime completely as oxalate, and after cooling the whole is run into a 200 c.cm. flask, and the volume made up to the mark with water. The oxalate of lime is filtered off, 100 c.cm. of the liquid taken, 10 c.cm. of concentrated sulphuric acid added, and the oxalic acid, not transformed into oxalate of lime, determined by titration with permanganate, from which the amount of lime which has remained on the filter as oxalate of lime is deduced.

2. *Sulphuric Acid.*—Beaume's areometer[1] will give a first indication of the strength of this acid, using Lunge and Isler's table. Sulphuric acid is estimated by a normal solution of caustic soda, the percentage of which has been tested in presence of phenol phthalein, for example, by a normal solution of pure oxalic acid at 63 grammes per litre, or by normal sulphuric acid at 49 grammes per litre. One hundred grammes of the sulphuric acid to be tested are weighed out, made up to a litre with water at the surrounding temperature, then 10 c.cm. are drawn off, a few drops of an indicator like phenol phthalein added and the normal soda solution run in. N being the number of cubic centimetres of soda added to cause the indicator to change colour, the percentage of sulphuric acid is got by the equation

$$N \times 0{\cdot}049 \times 100.$$

B. *Analysis of Finished Products:* 1. *Pyrolignite of Lime.*—A fair average sample is taken and prepared representing the bulk of the lot of pyrolignite of lime, of which an exact analysis is to be made. Several processes are employed to determine the strength of pyrolignite of lime: (1) 100 grammes of pyrolignite of lime are triturated in a test glass on foot or a mortar with boiling water; the liquid is filtered, and the lime precipitated in the filtrate by ammonium oxalate. The pre-

[1] Twaddell's hydrometer is invariably used in Britain.—Tr.

cipitate of oxalate of lime formed is filtered after a short time, then washed and dried; it is then moistened with nitric acid and calcined. The lime obtained is weighed after cooling. The amount of acetic acid necessary to saturate the lime found is determined by calculation, by multiplying the weight of the lime by $\frac{120}{56} = 2{\cdot}143$. As, by this process, it is taken for granted that all the lime which is dissolved in water is in the state of pyrolignite, the result obtained is a little too high, because, as already mentioned, pyrolignite contains free lime. (2) 100 grammes of pyrolignite of lime are treated with 90 grammes of commercial hydrochloric acid; the substances are left in contact for some hours, after which the liquid is distilled in a tubulated retort, fitted with a bromine tube and a condenser; when the distillation of the acetic acid appears to be finished, 100 grammes of water are added to the retort and the distillation and addition of water continued, so long as a liquid is collected having an acid reaction. The liquors A, the product of the distillation, are mixed and brought to a given volume, of which 10 c.cm., for example, are taken for titration with a solution of normal soda. As the hydrochloric acid added in excess distils with the acetic acid, a certain correction must be made, which is determined by the estimation of the hydrochloric acid contained in 10 c.cm. of solution A. This titration is carried out by exactly neutralising 10 c.cm. by a solution of pure carbonate of soda free from chlorine; a few drops of a saturated solution of neutral chromate of potash are then added, then from a graduated burette a standard solution of nitrate of silver is run in until the liquid becomes red; all the chloride of silver is then precipitated. In that way the amount of hydrochloric acid in solution A is determined, from which the weight of acetic acid contained in 100 grammes of the pyrolignite of lime is deduced. (3) 2 grammes of finely pulverised pyrolignite of lime are run into a 500 c.cm. flask, then 20 c.cm. of pure phosphoric acid of 45° B. (91° Tw.), sp. gr. 1·455. The flask is fitted with an india-rubber cork with two holes, through one of which passes a delivery tube, which conveys the distilled vapours to a condenser; through the other passes a tube drawn out to a point which, reaching near to the bottom of the flask, conveys steam into it produced in another flask. The acetic acid liberated by the phosphoric acid is entrained by the steam, and is carefully condensed, until the liquid which flows away is neutral to litmus. To attain that result, from eight to ten hours must be allowed for the operation and at least a litre of acid water must be collected; an acidimetrical titration and a simple calculation give the strength, in acetic acid, of the pyrolignite of lime tested.

This method is frequently adopted at the present time, owing to its exactitude, whilst at the same time it entails but little supervision. Frésénius has modified it as follows: (4) 5 grammes of pyrolignite of lime and 50 c.cm. of phosphoric acid 45° B. are run into a 200 c.cm. long-necked flask also fitted with a cork with two holes. The mixture is cautiously heated without passing steam, then the heat is increased and reduced afterwards as soon as the mass assumes a very pasty appearance. The whole is allowed to cool slightly and steam injected until the distillate has reached a volume of about 150 to 200 c.cm. To estimate rapidly the amount of lime in a pyrolignite moisten the pyrolignites lightly with nitric acid, then calcine in a muffle furnace, cool and weigh. This gives the total lime.

2. *Acetates other than Acetate of Lime.*—The acetic acid is estimated by one of the methods previously given, say, the third method.

TABLE XVI.—SHOWING THE PERCENTAGE OF ACETATE OF SODA IN SOLUTIONS OF THE SALT OF VARIOUS DENSITIES

Density.	Per cent.	Density.	Per cent.	Density.	Per cent.	Density.	Per cent.
1·0058	1	1·0488	9	1·0910	17	1·1440	26
1·0116	2	1·0538	10	1·0966	18	1·1506	27
1·0174	3	1·0591	11	1·1074	20	1·1572	28
1·0232	4	1·0644	12	1·1134	21	1·1638	29
1·0292	5	1·0697	13	1·1194	22	1·1706	30
1·0341	6	1·0750	14	1·1254	23	...	...
1·0390	7	1·0802	15	1·1314	24	...	...
1·0439	8	1·0856	16	1·1374	25	...	...

When it is a case of a solution of acetate of soda, the above table gives an idea of the strength of the solution. Finally, if it be desired to estimate the lead in an acetate of lead, it is estimated as sulphate. It suffices to calcine a known weight of the lead salt in a porcelain crucible, which is previously moistened with nitric acid, then by a few drops of sulphuric acid.

3. *Titration of Acetic Acid.*—The titration of acetic acid is effected by an acidimetrical test, either by volume or by weight. The French excise method *by volume* is as follows: From 50 to 100 c.cm. of the acetic acid to be titrated, diluted according to its strength with one, two or three times by volume of water, are run into a graduated 250 c.cm. flask. The solution is afterwards intimately mixed, and a known volume of it is then run into another graduated flask with a round bottom, to which two drops of a solution of phenol phthalein are added, then a normal solution of caustic soda is run into it gently until a rose coloration appears, taking care to shake after each addition

of soda. By a simple calculation the strength of the acetic acid is determined.

To estimate the acetic acid *by weight,* 50 grammes of this acid are weighed exactly into a 500 c.cm. flask, and the volume made up to the mark with distilled water, then 10 c.cm. are drawn off with a graduated pipette and run into a test glass, phenol phthalein added, and a decinormal solution of soda run in from a Mohr's burette, with constant stirring, until a rose coloration appears. By multiplying by 0·60 the number of cubic centimetres of soda solution required for neutralisation, the weight of acetic acid contained in 100 grammes is obtained. Mohr has given the following table of the densities of mixtures of acetic acid and water.

TABLE XVII.—SHOWING THE PERCENTAGE OF GLACIAL ACETIC ACID IN MIXTURES OF ACETIC ACID AND WATER OF VARIOUS DENSITIES (MOHR)

Per cent.	Density.	Per cent.	Density.	Per cent.	Density.	Per cent.	Density.
100	1·0635	82	1·073	64	1·068	46	1·055
99	1·0655	81	1·0732	63	1·068	45	1·055
98	1·067	80	1·0735	62	1·067	44	1·054
97	1·068	79	1·0735	61	1·067	43	1·053
96	1·069	78	1·0732	60	1·067	42	1·052
95	1·070	77	1·0732	59	1·066	41	1·0515
94	1·0706	76	1·073	58	1·066	40	1·0513
93	1·0708	75	1·072	57	1·065	39	1·050
92	1·0716	74	1·072	56	1·064	38	1·049
91	1·0721	73	1·071	55	1·064	37	1·048
90	1·073	72	1·071	54	1·063	36	1·047
89	1·073	71	1·071	53	1·063	35	1·046
88	1·073	70	1·070	52	1·062	34	1·045
87	1·073	69	1·070	51	1·061	33	1·044
86	1·073	68	1·070	50	1·060	32	1·042
85	1·073	67	1·069	49	1·059	31	1·041
84	1·073	66	1·069	48	1·058	30	1·040
83	1·073	65	1·068	47	1·056	29	1·039

When a very dark-coloured acetic acid has to be titrated, such as pyroligneous acid, for example, it cannot be done directly. In that case 10 c.cm. or 10 grammes of the acid to be tested are run into a small flask on to a known excess of barium carbonate weighed exactly. The whole is boiled; barium acetate forms which dissolves; the excess of barium carbonate is filtered and well washed; the filter and its contents are then placed in a beaker, with a few drops of tincture of litmus, then a standard solution of nitric acid, until the liquid is acid and evolves no more carbonic acid on heating. The solution is then neutralised by a standard alkaline solution. The total amount of carbonate of baryta which has not been dissolved by the acetic acid is determined in that way, from which the weight of the acetic acid contained

in the pyroligneous acid is calculated. In the case of concentrated acids, the properties which oil of lemons and poppyseed oil dissolved in acetic acid possess are utilised. Oil of lemons is easily dissolved by 94 per cent. acid in the ratio of 1 to 10, whilst poppyseed oil only dissolves in all proportions in more concentrated acid.

TABLE XVIII.—SHOWING CONGEALING POINT OF DILUTE ACETIC ACID OF DIFFERENT STRENGTHS

Water added to 100 Parts of Acetic Acid.	Percentage of Water contained in the Acetic Acid.	Congealing Point.	Water added to 100 Parts of Acetic Acid.	Percentage of Water contained in the Acetic Acid.	Congealing Point.	Water added to 100 Parts of Acetic Acid.	Percentage of Water contained in the Acetic Acid.	Congealing Point.
		°C.			°C.			°C.
0	0	+16·7	5	4·761	+9·4	12	10·774	+2·7
0·5	0·497	+15·65	6	5·660	+8·2	15	13·043	−0·2
1	0·980	+14·80	7	6·542	+7·1	18	15·324	−2·6
1·5	1·477	[illegible] 4	8	7·407	+6·25	21	17·355	−5·1
2	1·961	+13·25	9	8·257	+5·3	24	19·354	−7·4
3	2·912	+11·95	10	9·090	+4·3			
4	3·846	+10·50	11	9·910	+3·6			

4. *Analysis of Crude Wood-Spirit. Alcoholometry.*—The use of alcoholometers, say Gay-Lussac's, Richter's or Tralles', gives directly the approximate percentage of pure alcohol contained in crude wood-spirit.

TABLE XIX.—SHOWING THE DENSITIES OF MIXTURES OF ALCOHOL AND WATER CONTAINING, PER 100 VOLUMES, *n* VOLUMES OF ABSOLUTE ALCOHOL (*n* = DEGREES GAY-LUSSAC)

Alcohol Per cent.	Density.	Alcohol Per cent.	Density.	Alcohol Per cent.	Density.	Alcohol Per cent.	Density.	Alcohol Per cent.	Density.	Alcohol Per cent.	Density.
1	0·999	18	0·978	35	0·960	52	0·932	69	0·893	86	0·848
2	0·997	19	0·977	36	0·959	53	0·930	70	0·891	87	0·845
3	0·996	20	0·976	37	0·957	54	0·928	71	0·888	88	0·842
4	0·994	21	0·975	38	0·956	55	0·926	72	0·886	89	0·838
5	0·993	22	0·974	39	0·954	56	0·924	73	0·884	90	0·835
6	0·992	23	0·973	40	0·953	57	0·922	74	0·881	91	0·832
7	0·990	24	0·972	41	0·951	58	0·920	75	0·879	92	0·829
8	0·989	25	0·971	42	0·949	59	0·918	76	0·876	93	0·826
9	0·988	26	0·970	43	0·948	60	0·915	77	0·874	94	0·822
10	0·987	27	0·969	44	0·946	61	0·913	78	0·871	95	0·818
11	0·986	28	0·968	45	0·945	62	0·911	79	0·868	96	0·814
12	0·984	29	0·967	46	0·943	63	0·909	80	0·865	97	0·810
13	0·983	30	0·966	47	0·941	64	0·906	81	0·863	98	0·805
14	0·982	31	0·965	48	0·940	65	0·904	82	0·860	99	0·800
15	0·981	32	0·964	49	0·938	66	0·902	83	0·857	100	0·795
16	0·980	33	0·963	50	0·936	67	0·899	84	0·854		
17	0·979	34	0·962	51	0·934	68	0·896	85	0·851		

If the temperature be 15° C. + n it is necessary to deduct 0·4 n alcoholometric degrees in order to get the alcoholic strength. It is necessary to add them on the contrary if $t = 15 - n$.

Tralles' table differs slightly from that of Gay-Lussac. It gives the alcoholic strength at 15·56° C. Take T as the strength in degree Tralles and D the density at 15·56, and we get :—

TABLE XX.—SHOWING THE DENSITY OF ALCOHOL OF VARIOUS STRENGTHS ACCORDING TO TRALLES

T =	D =	T =	D =	T =	D =
0	0·9991	50	0·9335	85	0·8488
10	0·9857	60	0·9126	90	0·8332
20	0·9751	70	0·8892	95	0·8157
30	0·9646	75	0·8765	100	0·7939
40	0·9510	80	0·8631		

To get the percentage of alcohol *by weight* (x) from the percentage by volume determined by the alcoholometer (V), the density of the mixture in the table is taken (D) and that of pure alcohol 0·795, and the following calculation made, $x = V \frac{0{\cdot}795}{D}$. In Germany use is made of alcoholometers giving the percentage by weight at 15° C.

Determination of Methyl Alcohol.—This method consists in converting methyl alcohol into methyl iodide which is weighed. In a small flask 22 grammes of iodine are dissolved in 5 grammes of methyl alcohol to which its own bulk of water has been added ; the flask is corked, shaken and left for ten to fifteen minutes in a bath of cold water to bring it to the ordinary temperature, then 2 grammes of phosphorus are added and the flask is attached to an ascending condenser. About a quarter of an hour afterwards, the temperature of the water is gradually increased to 75° C., shaking the mixture from time to time ; the water bath is kept from a quarter of an hour to twenty minutes at that temperature, then it is allowed to cool. The methyl iodide formed is distilled, condensed by a descending condenser, and collected in a graduated cylinder. Working on 5 c.cm. of wood-spirit, the number of cubic centimetres of methyl iodide found, multiplied by 12·94, gives the volume per 100 c.cm. of methyl alcohol.

Determination of Acetone.—Several methods may be adopted in the estimation of acetone.

Kramer's Method.—This method is based on the conversion of acetone into iodoform by iodine in presence of an alkali. The wood-spirit is diluted so that it contains 1 per cent. of acetone. A rough test is first made. One c.cm. of wood-spirit diluted with water is run

into a flask with stopper with 10 c.cm. of binormal soda (80 grammes of NaHO per litre). The whole is shaken and 5 c.cm. of a binormal solution of iodine (254 grammes of iodine per litre) added. Iodoform is formed, which is shaken with 10 c.cm. of ether free from alcohol which dissolves it, then the volume of the ethereal layer is determined and 5 c.cm. of it taken and evaporated on a watch glass, placed in a desiccator containing sulphuric acid as a dehydrating agent. From the weight of iodoform found the percentage of acetone is calculated: 1 molecule of iodoform 394 = 1 molecule of acetone, or 58.

Messinger's Volumetric Method.—This method is based on the reaction described above, but with excess of iodine in alkaline solution, which is afterwards estimated by hyposulphite of soda, the solution having been previously acidified. The solutions used are: (1) A solution of sublimed iodine $\frac{1}{5}$ normal, say 25·4 grammes per litre, which is dissolved by double its weight of potassium iodide. (2) Solution of hyposulphite of soda $\frac{1}{20}$ normal, say 62·025 grammes pure air dried per litre, dissolved in distilled water, to which 15 c.cm. of caustic soda solution has been added. These two solutions should be strictly exact, that is to say 1 c.cm. of the standard iodine solution should correspond to 4 c.cm. of the standard hyposulphite solution. (3) Solution of *caustic soda* obtained by dissolving 1 kilogramme of caustic soda in flakes and the solution made up to 10 litres. (4) Sulphuric acid solution made from 275 c.cm. of sulphuric acid 66° B. (168° Tw.), sp. gr. 1·840, in enough distilled water to make 5 litres. (5) Starch paste made by stirring up 5 grammes of starch in 500 c.cm. of distilled water, boiling for an hour, then completing to 1 litre with salt water. To obtain good results, the product to be analysed should contain about 0·5 per cent. of acetone. By means of a pipette graduated in two places, 20 c.cm. of the liquid to be analysed is run into a 1 litre flask already containing a certain amount of distilled water free from nitrites or organic matter. The volume is made up to 1 litre, and the flask and contents well shaken to render the liquid perfectly homogeneous. Into a 250 c.cm. flask with a ground glass stopper, containing already 30 c.cm. of binormal soda, there are run 20 c.cm. of the dilute solution, the acetone content of which it is desired to ascertain. After mixing, 55 c.cm. of the $\frac{1}{5}$ iodine solution are added from a burette. The whole is again agitated and allowed to react fifteen to twenty minutes. Then 35 c.cm. of normal sulphuric acid are added, and the liberated iodine is titrated by hyposulphite of soda, which is run in from a graduated burette until the solution is almost decolorised. At this moment 4 to 5 c.cm. of starch paste are added, and hyposulphite of soda is run in until complete decolorisation is effected. Let N be the number of cubic centi-

metres of hyposulphite used, $\frac{N}{4}$ will correspond with the iodine in excess, from which the iodine combined with the acetone can be deduced. This amount of iodine multiplied by 0·6073 gives the percentage of acetone contained in the liquid to be estimated. Two parallel experiments are generally made, one with a typical methyl alcohol (methylene), obtained by adding 25 per cent. of pure acetone to pure methyl alcohol, the other with the methylene to be tested. When the operator has had no experience in this style of analysis, it would be well to perform the determinations in duplicate. This process is used in the laboratory of the French Minister of Finance.

Denigès' Method (*Journal de Pharmacie et de Chimie,* 1899, ix., 7) is based on the property of acetone, giving a crystalline precipitate with excess of mercuric sulphate, the formula of which is $[(SO_4Hg)^2\ 3HgO]^3\ 4CO(CH_3)^2$, which dried at 110° C. (230° F.) becomes $[(SO_4Hg)^2\ 3HgO]\ CO(CH_3)^2$.

Estimation of Allylic Alcohol.—This process is based on the property which allylic alcohol possesses of yielding an addition compound with bromine: one molecule of allylic alcohol absorbs two atoms of bromine. The solution of bromine used contains 7·3 grammes of bromine per litre; and it is prepared by dissolving in distilled water 2·447 grammes of potassic bromate and 8·719 grammes of potassium bromide, both dried at 100° C. (212° F.), and the volume made up to 1 litre; 100 c.cm. are taken for analysis, 20 c.cm. of sulphuric acid of 1·29 density added, to which mixture the wood-spirit is added until a persistent coloration is obtained.

Determination of the Impurities in Wood-Spirit.—The method used is that of Rose, which consists in noting the increase in volume which a known volume of chloroform undergoes when agitated with methyl alcohol in presence of a solution of sodium bisulphite. The apparatus used is a bulb tube (Fig. 62), the lower portion of which, of 55 c.cm. capacity, is graduated into tenths from 50 to 55 c.cm.; 50 c.cm. of pure chloroform at 15° C. are run into this tube from a burette with two marks, then a mixture of 25 c.cm. of the methyl alcohol to be tested and 60 c.cm. of distilled water. The whole is energetically shaken and allowed to stand in a bath of cold water at 15° C., then the increase in volume of the chloroformic solution is noted.

Estimation of Ethers in Methyl Alcohol.—This estimation is effected by a soda saponification, using 20 c.cm. of the methyl alcohol which is run into a 200 c.cm. flask with 50 c.cm. of semi-normal caustic soda and a few drops of a 1 per cent. alcoholic solution of phenol phthalein. The flask is connected with a reflux condenser and is boiled for half

an hour, then the soda in excess is titrated back with semi-normal sulphuric acid. The saponifiable products are calculated to methyl acetate by the formula

$$(50 - N) \times 0{\cdot}3894 \times 5,$$

N being the number of cubic centimetres of sulphuric acid used.

5. *Assay of Pure Methyl Alcohol.*—(1) The methyl alcohol should register 99 per cent. by the alcoholometer, say 0·7995 density at 15° C. (2) It must not contain more than 0·7 per cent. of acetone by Kramer's method. (3) On distillation 95 per cent. ought to pass between two consecutive degrees C. of the thermometer. (4) With double its weight of sulphuric acid of 66° B. (168° Tw.), sp. gr. 1·84, it should give at the most a pale yellow. (5) It should remain colourless with an ex-

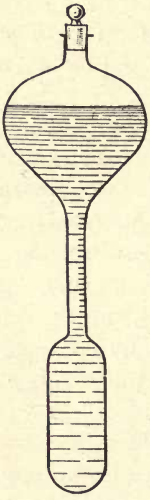

FIG. 62.—Rose's tube.

FIG. 63.—Reduction tube.

cess of caustic soda. (6) 5 c.cm. should not immediately decolorise 1 c.cm. of potassium permanganate of 1 gramme per litre.

6. *Assay of Acetone.*—(1) It should be limpid and pale. (2) It should mix without turbidity or eventually precipitate with water in all proportions. (3) The acetone should be neutral and mark 98·5 G.L. at 15° C. (59° F.) by the alcoholometer. (4) On distillation 95 per cent. of the liquid should distil at 58° C. (136·4° F.). (5) A solution of mercuric chloride should give no turbidity with acetone. (6) The acetone should not contain more than 0·1 per cent. of aldehyde, which is determined by reduction by a solution prepared from 30 grammes of nitrate of silver, 30 grammes of caustic soda and 200 grammes of ammonia (of 0·900 density), all dissolved in distilled water to 1 litre;

10 c.cm. of acetone are taken, its volume of distilled water and 2 c.cm. of the silver solution added ; it is left for a quarter of an hour in darkness, then it is tested by ammonium sulphide to see if there be nitrate of silver in excess ; if not the acetone contains more than 1 per cent. of aldehyde. (7) By Messinger's iodometric assay 98 per cent. at least should be obtained.

7. *Assay of Creosote.*—Beech-wood creosote being the most valued, impure products and even ordinary carbolic acid are substituted for it in commerce ; sometimes it is mixed with alcohol, or oily matter, fixed and volatile. All creosotes which run into water drop by drop, do not fall to the bottom, or which become turbid after slight agitation should be rejected. To distinguish carbolic acid from creosote or to detect its admixture with the latter, one of the following methods may be employed : (1) By mixing equal volumes of creosote and glycerine the latter is not dissolved, whilst carbolic acid dissolves completely therein and is not precipitated by the addition of water ; (2) ammonia does not dissolve creosote, whilst (when hot) it partially dissolves carbolic acid ; (3) a slightly ammoniacal alcoholic solution of perchloride of iron colours creosote green (by this reaction 1 part of creosote in 500 parts of carbolic acid can easily be detected) ; an aqueous solution of perchloride of iron added to creosote is not changed in colour, whereas it becomes blue with carbolic aid. To detect small amounts of carbolic acid in creosote, a few drops of the latter are boiled with 6 to 8 c.cm. of nitric acid until no more red fumes are given off, then a solution of potash is added when cool. If an immediate precipitate of picrate of potash is produced, picric acid has been formed at the expense of the carbolic acid existing in the creosote. To detect alcohol mixed with the creosote, the creosote is distilled and the first runnings are collected, or better, six times its weight of sweet almond oil is added to the product ; if there be only 0·4 per cent. of alcohol the mixture becomes opaque and remains so after agitation.

Fixed or volatile oils are easily detected. By pouring a little of the liquid on paper a spot will be formed, which remains transparent when cold if essential oil be present and does not change appearance under the action of heat in the presence of fixed oils. By adding to a known weight of the suspected creosote a sufficient quantity of acetic acid, the fatty substances insoluble in the acid may be isolated whilst the creosote dissolves.

8. *Analysis of Carbonate of Potash :* (1) *Moisture.*—Dry 10 grammes of the product over a bunsen burner until no water is given off and determine the loss in weight.

(2) *Insoluble Residue.*—Dissolve 10 grammes of carbonate of potash

in hot water, filter through a tared filter, make up to 500 c.cm. with the wash water ; dry the filter and contents, ignite and weigh the insoluble residue.

(3) *Alkalinity.*—Draw off 50 c.cm. of solution from 2 and determine the total alkalinity as potassuim carbonate by standard acid, using litmus as indicator.

(4) *Ponderal Degree.*—According to Gay-Lussac a solution of sulphuric acid is prepared thus:—

Sulphuric acid 66° B. (168° Tw.), sp. gr. 1·84 . . . 100 grammes.

Distilled water to bring the volume to 1 litre.

Weigh out a semimolecule of K_2O, say 48·07 grammes of the potash salt to be tested, which is dissolved in enough water to make 500 c.cm. From that solution 50 c.cm. are drawn off, tincture of litmus added, heat applied and the acid solution is run into it from a burette ($\frac{1}{2}$ c.cm. = 1 per cent. K_2O in the sample : *i.e.*, the ponderal degree).

Alkalimetrical Degree or Descroisilles' Test.—The test sulphuric acid is the same as the preceding. Five grammes of the potash salt are weighed, dissolved in water and heated, then litmus is added and standard acid run in from an alkalimeter with 100 divisions, each of which is equal to 0·500 gramme of the test liquor. The number of divisions indicates the alkalimetrical degree.

TABLE XXI.—FOR THE CONVERSION OF ALKALIMETRICAL DEGREES INTO PONDERAL DEGREES

Ponderal.	Alkalimetric.	Ponderal.	Alkalimetric.	Ponderal.	Alkalimetric.	Alkalimetric.	Ponderal.	Alkalimetric.	Ponderal.	Alkalimetric.	Ponderal.
1	1·04	9	9·36	45	46·81	1	0·96	9	8·65	45	43·26
2	2·08	10	10·40	50	52·01	2	1·92	10	9·61	50	48·07
3	3·12	15	15·60	55	57·21	3	2·88	15	14·42	55	52·88
4	4·16	20	20·80	60	62·41	4	3·85	20	19·23	60	57·68
5	5·21	25	26·0	65	67·61	5	4·81	25	24·03	65	62·49
6	6·24	30	31·20	70	72·81	6	5·77	30	28·84	70	67·30
7	7·28	35	36·41	75	78·01	7	6·73	35	33·65	75	72·10
8	8·32	40	41·61	80	83·21	8	7·69	40	38·46	80	76·94

Estimation of Soda in Potash.—By Graeger's process, which consists in dissolving 6·911 grammes in 100 c.cm. of water, collecting and weighing the insoluble residue, then in determining, volumetrically, in a portion of the solution the combined hydrochloric and sulphuric acids which are converted by calculation into potassium salts and the weight of the pure alkaline carbonates determined by difference. The carbon-

ates are then titrated by a normal solution of nitric acid (63 grammes HNO_3 per litre) corresponding to 69 grammes K_2CO_3. The ratio of the carbonates is given by the following table :—

TABLE XXII.—SHOWING THE NUMBER OF CUBIC CENTIMETRES OF NORMAL ACID REQUIRED TO NEUTRALISE 1 GRAMME OF A MIXTURE OF THE CARBONATES OF POTASH AND SODA IN VARIOUS PROPORTIONS

Grammes K_2CO_3.	Plus grammes Na_2CO_3.	Require Normal Acid in c.cm.	Grammes K_2CO_3.	Plus grammes Na_2CO_3.	Require Normal Acid in c.cm.
1·00	0·00	14·47	0·45	0·55	16·89
0·95	0·05	14·69	0·40	0·60	17·11
0·90	0·10	14·92	0·35	0·65	17·33
0·85	0·15	15·14	0·30	0·70	17·55
0·80	0·20	15·35	0·25	0·75	17·76
0·75	0·25	15·57	0·20	0·80	17·97
0·70	0·30	15·79	0·15	0·85	18·19
0·65	0·35	16·01	0·10	0·90	18·40
0·60	0·40	16·23	0·05	0·95	18·62
0·55	0·45	16·45	0·00	1·00	18·84
0·50	0·50	16·67			

The amount of soda contained in a potash salt may be determined by difference, the potash being estimated by one of the undernoted processes.

Estimation of Potash.—Three different processes may be adopted: (1) The platinic-chloride process; (2) Schloesing's perchloric acid process; (3) Carnot's volumetric process.

Platinic-chloride Process.—Five grammes of carbonate of potash are are dissolved in water containing hydrochloric acid, an excess of baryta water added, and the precipitate of sulphate, silicate and phosphate of baryta filtered off. A current of carbonic acid gas is passed through the solution, and the operation is terminated by boiling, so as to decompose the resulting soluble bicarbonate of baryta. The liquid is then filtered from the insoluble carbonate of baryta, into which the excess of baryta water has been converted. The liquid, to which hydrochloric acid has been added, is evaporated in a 40 to 50 c.cm. capsule, then platinic chloride is added drop by drop, and the whole evaporated on the water bath till the mass becomes pasty; it is then withdrawn from the fire, and a mixture of alcohol and ether, consisting of 9 parts of 85 per cent. alcohol and 1 part of 65 per cent. ether is added; the whole is triturated for some time to wash the potassic platino-chloride which is formed, and which is insoluble in this liquid, and the precipitate is collected on a dry tared filter. This method requires much care; the following, which is more rapid, is preferred. It consists in the immedi-

ate formation of the potassic platinic-chloride. One gramme of the carbonate of potash is dissolved in a little water, and hydrochloric acid added. The solution is concentrated, then platinum chloride solution (10 grammes per 100 c.cm.) added, until no further precipitation takes place; the whole is then evaporated; if the residue becomes white on the sides that shows that sufficient platinum chloride solution has not been added, and more must, in that case, be added. Then the alcohol and ether mixture is added, the whole filtered through glass wool (Fig. 63), and the precipitate washed with etherified alcohol, until there is no more platinum in the filtrate (rose colour with potassium iodide). The tube is dried in the water bath, then a current of hydrogen is passed through the interior, so as to transform the salts into metallic platinum which is weighed: $Pt \times 0{\cdot}4786 = K_2O$.

In this operation it is necessary to heat the tube gently, and to wait until it is full of hydrogen before bringing a flame near it. On the other hand, the sulphuric acid necessary to convert the carbonate of potash into sulphate is determined separately. The pure carbonate of potash is then found from the results obtained by calculation.

Estimation of Potash by Schloesing's Process.—Five grammes of the salt are dissolved in 40 c.cm. of water and nitric acid, and evaporated to dryness. The residue is taken up by 20 c.cm. of nitric acid and 20 c.cm. of water and filtered; ammonia is added in excess to the clear solution to precipitate iron and alumina, and the precipitate filtered and washed. The filtrate and wash waters are collected in a 100 c.cm. flask which is made up to the mark with water. Then 20 c.cm. are drawn off, to which an excess of barium nitrate is added to precipitate phosphoric and sulphuric acids. The filtered liquid is evaporated to dryness, then taken up by 5 c.cm. of nitric acid and again evaporated. This operation is repeated twice. After the last operation 15 c.cm. of a 10 per cent. solution of perchloric acid is added, and heat applied to expel the excess of acid. Perchloric acid is prepared by treating ammonium perchlorate in the hot state with aqua regia. The reaction is effected in a glass flask. A mixture of perchloric and nitric acid is obtained containing a little hydrochloric acid which is slowly evaporated on a sand bath to expel all HCl and a part of the HNO_3. The heat is withdrawn when white fumes of perchloric acid begin to come off. After having expelled the excess of perchloric acid added to the potash salt, the mass is moistened with water, then the whole is washed with 85 per cent. alcohol, to dissolve the perchlorates—other than potassium perchlorate—which are simultaneously formed. The potassium perchlorate is collected on a small filter, washed with 85 per cent. alcohol, then dissolved by hot water; the solution is evaporated to

dryness, and the weight of perchlorate found, multiplied by 0·3393, gives the percentage of K_2O.

Volumetric Estimation of Potash by Carnot's Process.—This method is based on the reaction which is produced when a potassium salt comes in contact with a bismuth salt and a hyposulphite. A perfect, well-defined double hyposulphite of potassium and bismuth is formed, soluble in water, but precipitated in the form of a yellow powder on the addition of alcohol. In carrying out the process, if, for example, a sample of potassium chloride (muriate of potash) has to be tested, 5 grammes of the substance are taken, dissolved in 3 or 4 c.cm. of water and 10 c.cm. of a solution of bismuth chloride, 10 c.cm. of hyposulphite of lime and 160 c.cm. of alcohol added. The yellow precipitate is formed. After ten minutes the precipitate is filtered and washed with alcohol and then redissolved in hot water, and the hyposulphite which it contains estimated. With this end in view, 5 c.cm. of hydrochloric acid are added to the solution and a few drops of starch paste. A solution of standard iodine is then added until the characteristic blue iodide of starch is produced. The solution of iodine is titrated in such a way that 1 c.cm. = 0·91 of potash working on the pure carbonate. The operation is conducted very rapidly, the double hyposulphite of potassium and bismuth being very easily decomposed. The solution of bismuth chloride is prepared by treating 100 grammes of basic nitrate of bismuth by hydrochloric acid and heating gently. The whole is allowed to cool, strong alcohol added, and after standing filtered and the solution is then made up to a litre. The solution of hyposulphite of lime contains 200 grammes per litre. The iodine solution consists of 56·96 grammes of pure iodine and about 75 grammes of iodide of potassium per litre : 1 c.cm. of this liquor corresponds exactly with 0·01 gramme of K_2O.

APPENDIX TO THE DISTILLATION OF WOOD[1]

THE DESTRUCTIVE DISTILLATION OF OLIVE OIL RESIDUALS

Wood waste from saw-mills, from factories for the extraction of dyes or of tannic acid from wood, and olive oil residuals are often used as fuel in the factories in which they are produced, whether to heat a stove or to raise steam. Ingenious furnaces with capacious grates and with steps or tiers and even gas generators have been patented with this end in view, but they involve great initial expense and frequent and costly enough repairs. Nevertheless, and in spite of this main use, as fuel and other secondary utilisations, to be referred to hereafter, the consumption of sawdust and olive residuum is feeble compared with their production; that is why it has been rightly attempted to distil these products. As to the importance of distilling olive oil residuum (*grignons d'olives*) the following is the report of a lecture by D. Vladimir Guerrero of Smirnoff. Manufacturers know by experience that hard woods give the best yield in alcohol, acid and charcoal, and that the latter is more dense than that got from soft wood. The charcoal obtained from olive residuum is still more dense than that from hard wood; the former weighs 37 kilogrammes per hectolitre (81·4 lb. per $2\frac{3}{4}$ bushels). That of the oak, which is one of the hardest varieties of wood, 22 kilogrammes (say 48·4 lb. per $2\frac{3}{4}$ bushels). That is due not only to its granular form but also to its richness in carbon, an exceptional richness verified by a greater number of calories, an observation already made by users of charcoal from olive oil residuum. The thermal value of one charcoal compared with another is verified by this: that the combustion of the

[1] The subject-matter contained in this appendix is in the original French edition interpolated in Chapter II., where its incorporation in the body of the chapter seems to the translator to not only break the continuity and disturb the logical sequence of the description of methods of carbonising wood given therein and of the plant employed, but it also causes the principles involved in destructively distilling olive oil residuals to lose half their force by being brought prematurely before the reader. The olive oil residual referred to consists essentially of the olive kernel and marc after extracting the oil by pressure.

most rich in carbon is, as is the case with charcoal from olive oil residuum, accomplished completely and regularly without production of carbonic oxide. The great density of olive oil residuum, like that of several other fruits, aids in demonstrating its great vascular richness and the purity of its carbon. It has been known from time immemorial that a very pure black, for fine carbon colours, was manufactured in Spain from peach stones under the name of Spanish black. The ligneous composition of the olive is essentially vascular. It, in fact, consists of: the almond, or seed, fatty, cellular and vascular; of the almost vascular kernel of the pulp, where the oil is found between the vascular and cellular tissue, and finally, of a hard, coriaceous skin, where a substance termed *cutose*, which contains carbon and vasculose in equal proportion, predominates. The vascular richness of plants depends chiefly on a dry climate and the direct action of the solar rays. Vasculose richer in *carbon* but poorer in *water* is a dehydrated ligneous body when compared with cellulose, which is consequently a hydrated ligneous body, and it may be added that cellulose is the product of a moist climate and vasculose of a dry climate. The varieties of olives cultivated for oil are more ligneous, and thus give a larger amount of residuum than those cultivated for preserves. The proportion of residuum, which is at least 18 per cent., and which may rise up to 30 per cent., is one-fourth of the bulk of the fruit. There are even varieties of olives in the province of Tarragona, for example, where the residuum weighs more than the pulp. Guerrero's experiments, verifying the previous investigations of eminent chemists on the kernels of certain fruits, have demonstrated that olive oil residuals contain the vasculose from which methylic alcohol and acetic acid have been extracted for about 100 years, and that it is a new source of raw materials for the pyroligneous industry. The wood wastes produced in several industries have been the subject of experiments, and more or less suitable plant for the extraction of pyroligneous products therefrom has been invented. Sawdust, which is very plentiful, has for a long time been utilised in wood distilleries, and it yields more or less interesting products according to whether it comes from a hard wood or a soft wood.

Bergmann's Patent.—Of late years it has been attempted to utilise the residuals from the extraction of tannic acid and dye woods. Bergmann's invention for this purpose made its appearance in Germany in 1892. It was applied. It gave no results, or rather it gave disastrous results. Some blamed the plant as badly thought out by its inventor. But that was not the cause. Bergmann's plant gave bad results for the simple reason that the raw materials could not cede what they did not possess, *i.e.*, they did not contain vasculose in the

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necessary proportion. Besides this reason there was another evil: the raw materials, very hygrometric, contain much water, 60 per cent., and with a great bulk and a great weight contained very little utilisable material.

Jurgensen's Patent.—Later on, Dr. Rolof Jurgensen, following without doubt in Bergmann's tracks (as the tracks of both are followed), brought out a process to treat all sorts of wood waste, including seeds and oleaginous fruits. Jurgensen, more fortunate than his predecessor, had the tact to distil olive oil residuum and obtained good results. He showed the products extracted therefrom [1] at the 1900 Paris Exhibition. Jurgensen, who experimented on large quantities, asserted that he had obtained very variable, but always satisfactory results. His process consists in treating olive oil residuum, such as it is, in a sort of extraction apparatus, but after previous drying, and for that he proposed to *press* them, being undoubtedly unacquainted with the method of treating olives. The carbonisation was effected in a plant (1900 pattern) intended to distil, in a continuous manner, not only sawdust but the tannic acid residues from the extraction of dyes from wood or from oil seeds.

Each woody substance has its own vascular richness and its different physical properties, and in some, these properties being diametrically opposed, absolutely different plant is required for their treatment.

Guerrero's modification of Jurgensen's Process as applied to Olive Oil Residuum.—When Guerrero took up the matter with Jurgensen, with the view of verifying the practical results which he had obtained in the distillation of olive oil residuum, he made certain objections to Jurgensen's process, which he believed to be capable of being advantageously modified. Accordingly, aided by the theoretical knowledge which is indispensable in practice, he sought to find out the process which should be followed, before that of the mechanical method, by which the process would be carried out. It was necessary to study the intimate nature of the substance, and the first thing which he had to do was to know the intimate composition of the different varieties of wood and of olive oil residuum. It was necessary to compare their density, bulk, shape, porosity and other physical and hygrometric properties, and likewise to know their chemical composition by starting from elementary analysis. It was also necessary to understand this industry, which is termed wood distillation. Guerrero therefore commenced, whilst visiting the different European factories, a series of experiments upon olive oil residuum—which he had caused to be sent to him from Spain—and upon several varieties of wood from

[1] Alcohol and acid.

different sources which he procured in the different factories which he visited; and then, placing himself in communication with the directors of the most famous laboratories of Paris, a plan was prepared for these investigations. Guerrero's researches are systematically expounded in that part of the memoir on the creation of the pyroligneous industry in Spain, which deals with experiments and analyses. Among other experiments made, attention has to be drawn to the comparative hygrometric properties (to which allusion has been made, as one of the causes of the poor success of Bergmann) of wood and olive oil residuum, which are as follows: Olive oil residuum without oil normally contains 12 per cent. of moisture, on account of its crushed condition, but it soon dries, as the water it contains is insignificant, olive oil residuum not being a hygrometric substance. On the other hand, wood, which is felled a year before distillation (for at the time of felling it contains 45 per cent.), still contains, when placed in the retorts, a fourth of its weight of water. This difference means an economy in distilling residuum as compared with wood. Comparative tests showed that olive oil residuum contains hard wood and soft wood, *i.e.*, cellulose and vasculose (the pulp, the hard skin and the *grignon*). This parallel established, and the properties of both substances being known, and also the advantage of carbonising vasculose rather than cellulose, an advantage which Guerrero early demonstrated, the facility of realising the distillation of olive oil residuum is a deduction therefrom. This inestimable advantage, impossible with wood, enables each substance to be treated appropriately. Guerrero, therefore, gave to the hard wood of olive oil residuum the name of *grignon riche* because it is rich in vasculose, and to the soft wood of olive pulp that of *grignon pauvre*. Such are the essential principles of Guerrero's process for extracting pyroligneous products from olive oil residuum. As to the mechanical system it could not be more simple; it is that of the coffee roaster. Olive oil residuum does not dry in the stove, as Jurgensen asserted. It is in vain to try it. It is dried in a sieve, by a fan, at the time it is separated into *grignons riches* and *grignons pauvres*. The *grignons riches* in the proportion of $\frac{4}{5}$ is the substance to be distilled. The residual *grignon pauvre* is an organic manure which, if need be, may be used as fuel mixed with tar.

Laboratory Experiments of Grandeau and Aubin.—The following are the results of numerous analyses and distillation experiments on olive oil residuum compared with wood :—

TABLE XXIII.—SHOWING THE AVERAGE PROXIMATE ORGANIC ANALYSES OF OLIVE OIL RESIDUUM AND WOOD

	Vasculose.	Cellulose.	Total.	Vasculose per cent.	Comparative Vascular Richness.	Comparative Ligneous Richness.
Wood (beech and oak) .	32·81	11·34	44·15	74·0	89	93
Rich kernel, 80 per cent.	36·83	10·45	47·28	80·0	100	100
Poor kernel, 20 per cent.	19·34	5·74	25·08	76·0	52	44

TABLE XXIV.—SHOWING THE AVERAGE RESULTS PER CENT. OF DISTILLING ASSORTED AND UNASSORTED OLIVE OIL RESIDUALS AS COMPARED WITH WOOD

	A.	B.	C.	D.	E.	F.
Methyl Alcohol . .	1·17	1·60[1]	1·20	1·38	100·0	86·0
Acetic Acid[2] . .	4·51	3·18	3·25	4·07	100·0	79·0
Tar	7·12	10·00	5·75	8·0	100·0	71·0
Charcoal . . .	29·00	37·00	25·50	31·7	100·0	80·0

A = *Grignons Riches* (Grandeau and Aubin). B = *Grignons Ordinaires*, without separating the *Grignons Riches*. Average factory results obtained in the Ivry-sur-seine wood distillery. C, Wood, average between pine, spruce and oak. D, olive oil residuum, average between the *grignon ordinaire* and the *grignon riche*. E and F, when olive oil residuum gives 100 lb. of each of the products, then wood yields the amount given under F.

If we compare the factory results in column D with those obtained from wood in the European carbonisation factories, the superiority of olive oil residuum is manifest. The pyroligneous yield of wood is less by one-fifth part than that obtained from the distillation of olive oil mill residuals. Such then are the results obtained as regards extracted products. The methyl alcohol and acetic acid obtained are the first got in the pure state from olive oil residuum.

Actual Working of Olive Oil Residuals. Extraction of the Oil from the Residuals. The Charcoal from the Residuals and its Applications.—The production of oil in Spain varies greatly from one year to another, so much that in certain districts it is almost *nil*, whilst in other and even neighbouring districts it is very abundant. For example, one year will give a produce of one, the following year of three, another year of five, or *vice versâ*, but the averages of each five years are almost the same. These differences of production are due, in the first place, to the natural condition of the plantations in dry soil, with no means in nine-tenths of the plantations for applying irrigation; they are, therefore, exposed to meteorological changes, which do not harmonise

[1] 7·38 of 20 per cent. phlegms. [2] Glacial.

very well with the rainfall, where these plantations abound. (A fresh proof of the want of forests.) These differences are also due to the poor regard we have for trees, which, like everything else, are grateful when well tended. Every one knows that this want of regard is due to a want of money, that is to say, of the working capital necessary for good farming, but very often this capital is there, and then it is the cultivation that is at fault, when it is not caused by excessive neglect. In a memoir presented to the Paris Exhibition of 1900, Manuel Porcer y Ruidor, who belongs to a family of olive-tree cultivators, where they appreciate the olive-tree, and where Guerrero knows by experience that the olive-trees bear fruit, estimates that the production of oil in Spain amounts to 2,900,000 hectolitres (63,800,000 gallons) in agreement with Dr. Monjarres of Seville. Guerrero believes that the production may, without exaggeration, be estimated at 2,700,000 hectolitres (59,400,000 gallons), by allowing for an extreme production of a fifth part, more or less, which would be 2,100,000 and 3,200,000 hectolitres (46,200,000 gallons and 70,400,000 gallons), which is equal to an annual average of 230 kilogrammes (506 lb.) per hectare (*i.e.*, per 2·47 acres), or say 205 lb. per acre. Consequently, the ratio of residuum to oil being known, there would be 409 kilogrammes of these residues per hectare, or say 364 lb. per acre per annum.

The figures given by Guerrero are those of 1898, a year when the production was inferior to the average; of thirty-three productive provinces, eleven only had a good yield. The total production was 1,900,000 hectolitres (41,800,000 gallons), of which 1,270,000 hectolitres (27,940,000 gallons) were for the districts of Andalusia and Estremadura. Three provinces, that of Jaen, Cordova and Seville, produced nearly 1,000,000 hectolitres (22,000,000 gallons), that of Jaen alone 500,000 hectolitres (11,000,000 gallons), say the fourth of the whole production. That district has the largest production in Spain, and it is to it that the others go to purchase the residuals for extraction. What is done with this enormous mass of woody fibre? Without counting exportation, which is insignificant, Guerrero estimates that 200,000 tons are utilised in the hundred odd oil mills, and that the rest is used as fuel; it is in fact the fuel used in the oil-producing countries, where the *grignon* replaces the scarce and dear wood. The oil residuals are employed for domestic purposes (kitchens and brasiers) in the manufacture of *grignon* charcoal, of lime, bricks and other industries. It is also used in the feeding of pigs and poultry; but frequently these residues are thrown into the stables. They lose in open air by slow combustion a third of their weight, without profit to any one. The 350,000 tons of oil residuals are produced on 1,150,000 hectares (2,840,500 acres), the

area under olive cultivation in Spain according to statistics. To find the forest surface necessary to produce this quantity of ligneous matter in the form of wood, such as oak, it suffices to know that this tree, arranged for felling at intervals of twenty years, produces in a good soil 35 tons per hectare (say 14 tons per acre) at the end of twenty years. Consequently, 350,000 tons of wood would require 10,000 hectares (24,700 acres), and for twenty years, one felling every year, twenty equal surfaces, say 200,000 hectares (say 500,000 acres). The value of olive oil residuum for the manufacture of pyroligneous products having been demonstrated, it follows that there are in Spain 200,000 hectares (500,000 acres) of virgin forest, much more productive than if it were planted with oak, which only wants a little initiative to exploit. The industry of extracting the oil from the residuals, with which, beyond the previous applications, no one knew what to do, has increased their value. With the installation of these factories they have risen (in Andalusia) from five pesetas, the price still current in some districts of the province of Jaen and other regions, to twenty and even thirty pesetas the ton, which is the average for five years. For several years the dry residuals were only used as fuel in the same factories, but large quantities remained piled up in the yards, up to the day when the idea came to convert them into charcoal known under the name of kernel or *grignon* charcoal. This manufacture became more general in proportion as the excellence of the new fuel became better known. This charcoal is now made in many Andalusian villages, and its production increases every year, as users prefer it to vegetable charcoal for many purposes. A few years ago *grignon* was sold with difficulty at 1·25 pesetas the hectolitre, $2\frac{3}{4}$ bushels; to-day whole waggons are charged with it for the North, or Madrid, for example, where it is sold at 5·50 pesetas the hectolitre.

In spite of that, coal, especially Andalusian, the calorific intensity of which varies between 4,500 calories and 6,500 calories, according to the proportion of ash, has been replaced by olive oil residuals in many industries, and the manufacturers who use this combustible would not change it for coal of average quality. The *grignon* in fact produces 4,500 to 5,000 calories when it is dry, but has the inestimable advantage of not making any clinkers. The experiments of Dr. Monjarres yielded still better results than those quoted. It follows from the latter experiments that olive oil residuals have a greater number of calories, by half, than wood, and that demands attention, because it confirms what Guerrero demonstrated by comparative analysis between these residuals and wood. Monjarres' experiments gave 5,270 calories for residuals and 3,600 for wood. The experiments quoted above, and the results attained in the

actual practice of this industry, confirm Guerrero's practico-scientific demonstrations, that is to say, the vascular riches of a residual neglected up to the present time, but which as a raw material for the production of methyl alcohol and acetic acid is without equal. It will be readily understood that the carbonisation of olive oil residuals, in closed vessels, would advantageously replace the distillation of wood, supposing that the wood distilling industry had been already successfully established in Spain, but there is no wood for rational working; on the contrary, however, vasculose, the raw material for that industry, exists in abundance in olive oil residuals. Of the actual production of residuals, rather more than a half is used to make *grignon* oil (*l'huile de grignon*); a very important amount still remains. If this utilisation continues, by combining oil extraction and carbonisation, a great saving would be made, because the cost price of the methyl alcohol and acetic acid, as well as their compounds and derivatives, would be very appreciably reduced. An idea of cost may be got by taking into account the fact that the residuals give a greater yield in acetic acid and in wood-spirit, that labour is cheaper than here, and that the raw material, the de-greased olive oil residual, costs less, especially when oil extraction and carbonisation, the most rational way of working, are done in the same factory. What competition is to be feared since wood costs at least twenty francs (sixteen shillings) the ton? If the enormous quantity of rich ligneous material contained in olive oil residuum could be distilled very quickly, that would occasion a crisis in the pyroligneous product market. That outlook is, however, not to be feared, because the consumption of these products increases daily owing to the developments of industry, and because this exploitation of olive oil residuals cannot become completely developed for rather a long time. In any case, the advantage would be, for Spain, that there would be no need to import either acetic acid or acetates, and if need be Spain could export its products, which need not fear any competition so long as acetic acid and methylic alcohol continue to be made from twenty-year-old oaks and beeches. The riches which would be created with the new profit of these residues is of capital importance for Spain.

At the present prices (the cost diminished by $\frac{1}{5}$) the extraction of oil would represent 26,000,000 pesetas, carbonisation 34,000,000, and between the two one-third the value of the oil, supposed to be only 180,000,000, calculating on an average of 2,700,000 hectolitres, or 243,000 tons of olive oil, at 750 pesetas the ton. Looking to the importance of wood distillation and the favourable conditions for establishing this industry in Spain with olive oil residuals, it may be asked "Why has it not been done before?" To which it may be replied

"Because the time had not come for it to be done". But another explanation must be forthcoming. The industry proper only flourishes in well-wooded countries because the raw materials are abundant there. It is, moreover, natural that residues which are only produced in large enough quantities in Spain should be passed by unseen. That is also explained by the insignificant consumption of pyroligneous products in Spain, compared with others. Then, more especially, the residuals are used for the extraction of industrial oils; and because of the methodical nature of the Spaniard, who has little inclination for new industries, this riches, like many others, has been passed by unseen. Owing to the intense felling of forests to supply the pyroligneous industry and its derivatives, as well as for paper manufacture, the price of wood, which continues to rise, increases the price of its products in proportion as their uses increase. At the 1900 International Congress of Applied Chemistry, held at Paris, Dr. Jurgensen urged that investigations be undertaken so as to place other raw materials at the service of wood distillers, especially cheap waste products, which could be used to produce acetic acid more cheaply. Dr. Jurgensen might have added that such new working-up of waste products would harmonise with the planting of forests (somewhat advanced in his country), because the industry, which consists in felling twenty-year-old trees, appears a little barbarous. This desire is exactly realised in Spain, where the olive produces a residue rich in vasculose, a material which, as Guerrero has demonstrated, is the best that could be chosen, and which at the same price might advantageously compete with wood. A triumph might thus be realised by Spain in the battles which industrial nations wage against one another. A good opportunity presents itself for following up the industrial reawakening of Spain, quite remarkable these four last years, as regards agricultural, chemical, metallurgical and other industries which have been installed without the aid of foreign capital. It is to be hoped that the new industry, essentially a chemical one, will very soon be inaugurated, and assist much to create those large chemical factories in Spain which give so much supremacy to Germany. Numerous chemical industries can be made to live in Spain. Amongst others, the manufacture of colours, margarine, glycerine, the manufacture of soda, and thus utilise numerous residues now almost all thrown away. Everything in Spain favours the creation of the new industry, and chiefly the progressive imports and the rise in the rate of exchange which financiers seek in vain to reduce. The attempts to create new industries, and the help which the Government ought to give to these, especially at the commencement, will be more efficacious in reducing the rate of exchange than financial speculations. Industries

will give work and bread, forthwith, to many poor people in the prosperous olive districts, but none the less poor in spite of that. Many other industries, anæmic up to now, would embrace this new industry, coming to give them an energetic push forward. How welcome it would be! The value of olive oil residuals is fully demonstrated. Its abundance is very great; it will be greater every day with the culture of the olive in new plantations. The pyroligneous products which can be extracted from it are of equal importance to those from wood. Notwithstanding the new industry, the cost of olive oil residuals for oil extraction will not increase, because use is now only made of 200 metric tons. By combining extraction with carbonisation, the cost price can only be less, and that of the pyroligneous products also. The cost price of methyl alcohol and acetic acid from olive oil residuals is one-fourth of that extracted from wood.

The consumption of pyroligneous products, which increases in every country, increases also in Spain. Methyl alcohol is destined to be the most important of the products, owing to the industrial applications of grain alcohol, and of the new industries which use methylic alcohol. The exploitation of pyroligneous products once increased, its use in Spain would be profitable, looking to the cost price of alcohol and acid from olive oil residuals. The creation of this new industry will increase the value of oil products. Manufacturers have a very lucrative road to a great future. The financiers of this enterprise, worthy of great initiative, can make of it a national industry. If Spain aspires to sustain its credit abroad, if it be desired to lower the rate of exchange, it is necessary to create new industries like this, which converts the abundant raw material and which after a little would increase her exports. Spain, in spite of everything, must follow the indefatigable march of progress; she must exploit this industry herself before others come to do it.

Vladimir Guerrero patented in September, 1905, a process by which he obtains the products derived from the distillation of olive oil residuals by two processes, separate or combined. The first is supplementary to the extraction of oil from the residuals, the second is for making chemical products by treating the ammoniacal liquor.

TABLE XXV.—PRODUCTS OBTAINED BY DISTILLATION OF 1 TON OF OLIVE OIL RESIDUALS AND THEIR VALUE

Products from Residuals.	Products from Ammoniacal Liquor.
Yield per ton :—	Minimum yield per cub. m. (220 gals.) :—
Granulated charcoal, 25 per cent. 11	Ammonium sulphate, 3·2 per cent. 14
Tar special, 3 per cent. . . 6	Methyl alcohol, 1·6 per cent. . 20
Ammoniacal liquor, 50 per cent. . 10	Acetic acid, 2·5 per cent. . . 36
Pesetas 27	Pesetas 70
At the rate of exchange 30 per cent., francs 20·75 (or 16*s.* 8*d.*).	At the rate of exchange 30 per cent., francs 53·85 (say 42*s.* 10*d.*, or 2¼*d.* per gal.).
Rich gas (light or power) : 160 cubic metres which produce 10 h.p. effective hour which, at 5 centimes each, represents 12 pesetas or 9·20 francs extra (7*s.* 4*d.*).	

These results are the fruit of three years' experiments made with a demonstration plant situated at Cordova, where Guerrero places himself at the disposal of manufacturers interested in his process and for which he is in a position to guarantee the yields given above. It is pleasant to register such a success, which is a great step in advance for this industry, the experimental field of which is very wide.

APPENDIX

DENSITY OF MIXTURES OF PURE METHYLIC ALCOHOL (WOOD-SPIRIT) AND WATER (DITTMAR AND FAWSITT)

Weight per cent.	Density at 0° C. (32° F.).	Density at 15·56° C. (60° F.).	Weight per cent.	Density at 0° C. (32° F.).	Density at 15·56° C. (60° F.).
1	0·99806	0·99729	63	0·90276	0·89133
2	0·99631	0·99554	64	0·90056	0·88905
4	0·99299	0·99214	65	0·89835	0·88676
6	0·98990	0·98893	66	0·89611	0·88443
8	0·98701	0·98569	67	0·89384	0·88208
10	0·98429	0·98262	68	0·89145	0·87970
12	0·98171	0·97962	69	0·88922	0·87714
14	0·97926	0·97668	70	0·88687	0·87487
16	0·97689	0·97379	71	0·88470	0·87262
18	0·97459	0·97039	72	0·88237	0·87021
20	0·97233	0·96808	73	0·88003	0·86779
22	0·97007	0·96524	74	0·87767	0·86535
24	0·96780	0·96238	75	0·87530	0·86290
26	0·96549	0·95949	76	0·87290	0·86042
28	0·96310	0·95655	77	0·87049	0·85793
30	0·96057	0·95355	78	0·86806	0·85542
32	0·95783	0·95053	79	0·86561	0·85290
34	0·95500	0·94732	80	0·86314	0·85035
36	0·95204	0·94399	81	0·86066	0·84779
38	0·94895	0·94055	82	0·85816	0·84521
40	0·94571	0·93697	83	0·85504	0·84262
42	0·94239	0·93335	84	0·85310	0·84001
44	0·93911	0·92975	85	0·85055	0·83738
46	0·93575	0·92610	86	0·84798	0·83473
48	0·93229	0·92237	87	0·84539	0·83207
50	0·92873	0·91855	88	0·84278	0·82938
51	0·92691	0·91661	89	0·84015	0·82668
52	0·92507	0·91465	90	0·83751	0·82396
53	0·92320	0·91267	91	0·83485	0·82123
54	0·92130	0·91066	92	0·83218	0·81849
55	0·91938	0·90863	93	0·82948	0·81572
56	0·91742	0·90657	94	0·82677	0·81293
57	0·91544	0·90450	95	0·82404	0·81013
58	0·91343	0·90239	96	0·82129	0·80731
59	0·91139	0·90026	97	0·81853	0·80448
60	0·90917	0·89798	98	0·81576	0·80164
61	0·90706	0·89580	99	0·81295	0·79876
62	0·90492	0·89358	100	0·81015	0·79589

PART II

THE MANUFACTURE AND TESTING OF TAN WOOD EXTRACTS AND THEIR UTILISATION IN MODERN TANNERIES

CHAPTER I

CHESTNUT EXTRACT

General Properties of Chestnut Wood—The Percentage of Tannin—Present Price of the Wood in France and Corsica—Deafforestation Yield in Extract of Wood from different Sources—The Water to be used in the Diffusion or Maceration of Wood—The Juice and Various Extracts from Chestnut Wood—Composition and Analytical Table

The chestnut is a tree of the family of Cupuliferæ ; it grows in Savoy, Auvergne, Périgord, Provence, Brittany, Corsica, to a small extent in the North of Spain and in Italy, but, as will be shown, is everywhere becoming scarcer. Chestnut wood has a certain amount of importance from the point of view of its tannin content. It was Michel of Lyons who discovered, in 1818, the presence of tannin in the chestnut-tree ; it was he, also, who suggested its use for the tanning of heavy leather. Chestnut wood intended for tanning, or for the manufacture of extracts for tanneries, should contain neither rotten wood, nor wood dead upon the tree, nor branches of less than 4 inches diameter at the small end, nor running roots. These restrictions do not apply to chestnut wood intended for the manufacture of extracts or gallic acid for dyeing. The cubic metre or stère of dry chestnut wood weighs 340 to 385 kilogrammes. The authors draw the attention of manufacturers of wood extracts and tanners, consumers of chestnut wood (which brings during the tanning of leather its contingent of pectic, resinoid and amylaceous matters favourable to a good yield), to the importance of always requiring from the suppliers deliveries of sound chestnut wood and rejecting rigorously rotten or ferruginous rotten wood, as incapable of a normal yield,

or only taking delivery of it with a heavy reduction in price. This damaged wood is only capable of being used for the manufacture of "gallic" acid for dyeing, the decolorisation of the liquor from the maceration of this rotten wood being difficult and costly.

TABLE I.—ANALYSES OF SOUND AND UNSOUND CHESTNUT WOOD FROM VARIOUS SOURCES

	Gard (Southern France).		Lyonnais and Dauphiné.	
	Sound, per cent.	Rotten, per cent.	Sound, per cent.	Rotten, per cent.
Tannin, soluble .	7·40	2·80	6·10	3·40
Non-tannin . .	1·90	1·10	1·50	1·00
Water . . .	54·25	63·28	53·20	64·24
Woody fibre . .	36·45	32·82	39·20	31·36
	100·00	100·00	100·00	100·00
Total soluble . .	9·30	3·9	7·60	4·4

The percentage of water being 54 per cent., that of the soluble tannin is then 4·08 per cent. The effect of the rotting of wood is to render insoluble a large proportion of the bodies originally soluble. As to the price of chestnut wood, it has done nothing but increase for the last five years, for reasons to be explained later on. In 1900 the extractors of the South of France and of Ardèche were already paying 14 to 15 francs (say 11s. to 12s.) the metric ton on the spot; at the present time they are paying 17 to 18 francs (13s. 7d. to 14s. 4d.). The price will go up still further as the chestnut plantations disappear daily and the country labourer is obliged to go farther afield to fell the trees, even to localities where the cost of felling, etc., of the metric ton of wood rises from 2 to 5 francs (say from 1s. 6d. to 4s.). In Corsica the extractor buys chestnut wood by the stère (about 460 kilogrammes) (say 1,012 lb.) at the rate of 9½ francs, say more than 20 francs (16s.) the ton. The freshly felled wood contains 68 per cent. of water; the green wood, three months after felling, contains 55 per cent.; the peeled wood 40 to 45 per cent. The wood is generally piled in stacks of 200 to 500 tons to dry, and the stock for consumption is made up from year to year. It loses on an average after that lapse of time 20 per cent. of water, and then only contains 33 to 35 per cent. The green wood of the chestnut-tree, with 75 per cent. of water, contains 4 per cent. of tannin on the trunk; ordinary wood, with 40 per cent. water, contains 6 per cent., and perfectly dry wood 8 per cent. Chestnut roots contain 7 per cent. of tannin; the bark 3 per cent. During

the drying of the chestnut wood a portion of the tannin resinifies. The northern chestnut wood is less rich than that of the south, and that of the west poorer than that of the east. The Corsican chestnut wood contains on an average 10 per cent. of tannin. Chestnut wood yields 4·74 to 5·71 per cent. of ash containing 70 to 80 per cent. of lime, 5 per cent. of potash and 4 per cent. of phosphoric acid. Its density is 0·588. The interesting question at the present time, and one which should engage the serious attention of the extract manufacturer and the large number of country folk, in whose life the chestnut plays an important part, is the reafforestment of the chestnut plantations which have already disappeared and those which will rapidly disappear. The numerous extract factories are as heedless of their future supply as the peasants, who fell the chestnuts, without concerning themselves about the future and who only see in this felling the source of present gain.

The Disappearance of the Chestnut-tree in France. The Cause and its Remedy.—During the discussion of the French budget of 1904 the Hon. M. Pedebidou on two occasions drew the attention of the Minister of Agriculture to the disappearance of the chestnut-tree and asked the Government to take steps to remedy such a disastrous situation. "The disappearance of the chestnut-tree is exerting a most vexatious influence on the regular flow of streams of water. The denudation of the slopes induces torrents, with injurious results to the navigation of rivers." But that is not all. The chestnut-tree is a very precious tree, the culture of which represents a valuable source of profit to many departments. In addition to its fruit, which constitutes an important article of food, it yields wood for making vine props and the hoops and staves of casks.

By trituration, it yields tannic extracts, the use of which is becoming more and more general. Essentially a hardy tree, the chestnut-tree adapts itself to the most intractable soils.[1] Its disappearance would constitute a real disaster. Grave uneasiness, therefore, is produced by the crisis through which this tree is passing. An inquiry was made by the Department of Rivers and Forests (*Eaux et Forêts*) on the cause of the destruction and the means of remedying it. The following are the conclusions.

The causes of the destruction of the chestnut are of two kinds: (1) The first consists in the chestnut disease, which assumes two distinct forms. One is the disease of exhaustion or decrepitude, which prevails in a great number of chestnut plantations, where the trees are old, because the cultivators impoverish them by stripping

[1] Chatin (Ad.), "Le Châtaignier, Étude sur les Terrains qui Conviennent à sa Culture," *Bull. de la Soc. Bot.*, p. 198, 1870.

off their green leaves, the only manure of the soil, and mutilate them by breaking and carrying away the branches, so as to procure wood for fuel and leaves for cattle-feeding. This disease is by no means of an epidemic nature, and attacks individual trees. To remedy it, all that has to be done is to stop the practices which occasion it, to preserve carefully the dead covering of the soil, to restrict the abuse of pasturage and to manage and exploit the chestnut in a rational manner, felling the old and replacing them by young trees. The second disease, which is the real chestnut disease, the ink or foot,[1] attacks all trees without distinction, young and old, vigorous or decrepit. It has a decidedly epidemic character, which has earned for it the title of phylloxera in certain districts. The seat of this disease is in the roots, and its origin is due to a new parasitical fungus, the *nycelophagus castanæ*. This disease has been reported from twenty-seven departments, in ten of which it has not made any considerable ravages as yet; eight showed depredations of less than 50 hectares (say 125 acres); in the following five, Dordogne, Gard, Ille-et-Vilaine, Morbihan, Lot, the area oscillates between 200 and 500 hectares (say 500 to 1,250 acres). Finally, the devastation does not reach 1,000 hectares (say 2,500 acres) in the Haut-Pyrenees, but it exceeds this figure in the Basses-Pyrenees, La Corrèze and La Haute-Vienne.[2] The devastated area amounts to about 10,000 hectares (25,000 acres) on a total area of 350,000 to 380,000 hectares (say 875,000 to 950,000 acres), not including the chestnut-tree coppices, about 100,000 hectares (say 250,000 acres). No economical and practical method has been found for coping with this disease; the only way of preventing the spread of the disease is to pull up the trees attacked. The chestnut-tree is liable to two other diseases, the jaundice and the quittor (*le javart*),[3] but happily these diseases are not widespread and rarely cause the death of infected trees.

(2) The most important cause of destruction consists in felling the chestnut-trees for the manufacture of tannic acid extracts. The use of tannic extracts tends to replace that of oak bark in tanning to an ever-increasing extent. Chestnut wood, the percentage of tannin in which is 4 to 8 per cent., yields in France the largest quantity of these extracts. In view of this new outlet, which enables this wood to command

[1] De Seynes (J.), "De la Maladie des Châtaigniers appelée Maladie de l'encre," *Comice Agricole de l'Arrondissement du Vigan*, September, 1889.

[2] Delacroix, "La Maladie des Châtaigniers en France," *Bull. de la Soc. Mycol. de France*, t. xiii., 4 fasc., 1897.

[3] Prilleux (Ed.), "La Maladie des Châtaigniers dite le Javart," *Journ. d'Agric. Pract.*, p. 139, 1893.

remunerative prices, landowners, for the sake of immediate gain, do not hesitate to fell immature chestnut-trees. They are encouraged in their work of destruction by a large number of factories for the manufacture of tannic acid extracts, which have been established in numerous districts, the needs of which are always increasing. It is, more especially, within the last twelve years that these factories have multiplied rapidly. In 1875 there were only seven. At the present day there are in France twenty-six factories exploiting the chestnut-tree for the extraction of tannic acid. To these factories must be added those of Geneva (Switzerland), which are supplied, for the great part, with French wood. The actual consumption of chestnut wood by these twenty-six factories may be estimated at 450,000 metric tons, producing extracts of a value of 22,000,000 to 24,000,000 francs (say £880,000 to £960,000). The hectare of chestnut-trees bears on an average a hundred trees (say forty trees to the acre), which may yield 375 tons of wood (say 150 tons to the acre); it will thus be seen that the consumption of chestnut wood by the tannic acid extract factories represents the disappearance of 1,200 hectares (say 3,000 acres) of chestnut-trees annually. This destruction is particularly intensive in Corsica, where it amounts to 200 hectares (say 500 acres) annually. In each of the departments of Gard, Lot, Dordogne, Corrèze, Haut-Vienne, De la Creuse and D'Ille-et-Vilaine, the area from which the chestnut has been felled now exceeds 1,000 hectares (say 2,500 acres); it is 500 to 1,000 hectares (1,250 to 2,500 acres) in the Basses-Pyrenees, La Lozère,[1] Saône-et-Loire, Loire Inférieure, Morbihan and the Côtes-du-Nord.[2] Finally, it amounts to 200 hectares (500 acres) in La Sarthe, L'Allier, Le Rhone, L'Isere, La Savoie, La Haute-Savoie and the Hautes-Pyrenees. Against the improvidence of the proprietors, which leads to this disastrous state of affairs, the Government is powerless. No legislative enactment can limit the right of the proprietor to use and abuse his chestnut plantations, except in very rare cases, provided for by the law relating to the reclamation of waste land. But the duty of the Government is to enlighten the rural population as to the gravity of the situation, and to make them understand the interest which they have in preserving and rationally exploiting the chestnut-trees. Formal instructions have been addressed to prefects, the agents of the Department of Rivers and Forests and the professors of agriculture, that they use all the means in their power to promulgate in country districts notions of

[1] Crié, "Rapport sur les Maladies des Châtaigniers dans les Cevennes," *Bull. du Minist. de l'Agric.*, No. 7, an xiv, October, 1895.

[2] "Rapport sur les Maladies des Châtaigniers en Bretagne," *Bull. du Minist. de l'Agric.*, No. 8, 1894.

prudence and wise foresight on this subject. On the other hand, where the destruction is already consummated, the replanting of the chestnut woods is to be encouraged. With this end in view, measures have been taken this year to award medals and prizes at the agricultural shows, so as to recompense work of this nature. Whilst approving the initiative taken by the Forest administration, in view of the systematic destruction of chestnut-trees for tannic acid extracts, more radical measures may be necessary to circumscribe or limit this destruction. Would not the best method of inducing proprietors to replant their chestnut woods be to award them a bounty, which would be debited against the factories themselves? It would suffice for that purpose to place a special tax, which need not be very high, on tannic acid extracts, per 100 kilogrammes, produced by the twenty-six factories already in existence. Nevertheless, so as to safeguard to a certain extent the interest of the tanner, and the very prosperous industries connected therewith, there would seem to be occasion to put a higher tax on foreign products imported for use in the tannery. A Bill, brought before the French Parliament on 6th July, 1889, by Dunaine and Hubert, complied to a certain extent with that object, since it tended to modify the general tariff of the French customs in regard to dye woods of foreign origin (quebracho and other woods). That Bill has never been discussed. But the attention of those interested is drawn to it. Again, as regards Corsica, a lecture of the 9th September, 1901, on "The Felling of Chestnut-trees in Corsica," by Donati, special Professor of Agriculture at Bastia, goes thoroughly to the root of the question.

"The question which I have to discuss before you is assuredly the most important of all those which concern not only the rural economy but all the well-being of Corsica. The destruction of chestnut-tree plantations by contractors who supply the tannic acid factories is pursued under such conditions that we consider it our duty to make known not only the advantages which the culture of the chestnut presents for our people, but also the causes of this deafforestation and its immediate consequences, not only from the point of view of food supply, but also as regards the climatology and the future of agriculture. These trees, which cover 30,000 hectares (75,000 acres) on the hill slopes, have always played an important rôle in all the critical periods of the history of Corsica; at the time when the people fought for their independence, they procured the means of existence from these trees; even at the present day, when misery extends its ravages farther and farther into the country districts, they constitute the principal resource of those farmers who have given up the cultivation of the soil. By its

richness in nutritive elements, the flour of the chestnut forms a complete food. The recent analyses by Balland (*Pharmacien Principal de Premiere Classe*) afford very precise data on this point: a lot of chestnuts from Piedmont, which are those which most resemble Corsican chestnuts, gave, on analysis, the following composition: albuminoids 5·98, fat 3·78, sugar and starch 86·82 per cent. If we compare these figures with the analysis of Dattel wheat, which is grown very widely, an analysis which is also given by Balland, it will be surprising to see how great the analogy is between these two products: albuminoids 10·53, fat 1·44, sugars and starch 84·28 per cent. The percentage of nutritive elements in chestnut approaches very much to that of wheat, and this explains how our highland population from time immemorial have been able to nourish themselves almost exclusively on soup and cakes made from chestnut flour. That shows us also the great benefit that may be drawn from these fruits in grave circumstances for the feeding of towns and villages. Chestnut wood is not less precious for domestic uses; exploited as coppices of five to six years old, it is used to make baskets, forks, whip-handles, tool-handles; it is used like the nettle-tree (*micocoulier*) and the willow (*osier*) in the south of France; as it gets bigger it is used as vine props and as wood for fences; used for carpentry purposes, it equals oak in durability; transformed into beams, planks and boards it is used for all the purposes of coach and waggon-building, cabinetmaking, etc., and even exposed to the wind, sun and rain, without being protected by any coating, its durability is unlimited; split up into staves it is used to make excellent vessels, of all dimensions, for the preservation of wine. As dead or withered wood it is equally useful, for it is an excellent firewood for ovens as well as for heating apartments. The cares required for the growth and management of these trees as well as for the collection of the fruits are almost *nil*. Generally the tree is a wild one—7 to 8 years old—which has come up naturally and been grafted on the spot; it grows as well as it can, surrounded by brushwood, until it commences to produce about the fifteenth or the twentieth year. The brushwood is then cleared away all round the plant, and after that the ground is cleared every year to allow the chestnuts to be collected. A chestnut may attain very great dimensions; many are met with which measure 6 and 7 metres (say 20 to 22 feet) in circumference and 25 to 30 metres (say 80 to 120 feet) in height. The value of these trees is based on their dimensions and the quality of their fruit; it varies from twenty to fifty francs (16s. to £2); some are worth more than a hundred francs (£4), but these are exceptional. The yield per foot easily reaches 10 to 12 per cent. of this

sum. The collection of the nuts is done, generally, by the proprietors themselves, and in that case it entails no great expense, because, with the exception of clearing away the brushwood and moss, which is done by men in their spare time, the nuts are gathered by women and children. When the landowners engage workpeople for the whole nut harvest they are paid in kind, say one-third or one-half. They, on their part, are bound over to proceed in the beginning of September with the clearing of the chestnut woods, to split up and carry the wood intended to feed the drying kilns, collect the nuts, carry them to the kilns, and dry them. The harvest requires about two months' work ; it commences from the 15th to the 20th of October and finishes about the end of November ; decortication is proceeded with as soon as the nuts are sufficiently dry to be brought to the mill. There is reason, therefore, to say that the chestnut is a most important tree ; and the void which the disappearance of these trees will create in country districts will be irreparable. The effect of this felling on the climate of the country will not be less disastrous. At the present time it pleases every one to extol the climate of Corsica. The town of Ajaccio owes to it the favour which it enjoys as a winter resort, but it is greatly to be feared that this will not be so when the slopes of the mountains are stripped, because it is recognised that it is the forests and the woods which render climates more genial, less variable and more constant. On the other hand, the influence of these forests is intimately connected with the prosperity of agriculture. They regulate the flow of torrents ; they retard the melting of the snow, and the water penetrating into the soil converts it into huge reservoirs yielding their overflow to the rivers even during summer as required. Hence the regularity in flow of rivers rising in wooded districts, whilst on bare ground the water rolls on in torrents carrying sand and gravel in its train, giving rise in the plains to floods which devastate the harvest and make considerable ravages. Besides the torrents from the hillsides are very abundant in winter and dry up in the heat of summer. The farmers of the arrondissement of Bastia have already had a foretaste of what is in store for them, on the night of the 4th August, 1899, when the waters of the rivers Alesani, Fiumalto, Golo and Bevinco overflowed their beds, and did considerable damage. These recent inundations cannot, it is true, be imputed to tree felling ; but these catastrophes, which up to now only reoccur at somewhat long intervals, will become more frequent owing to the destruction of the chestnut plantations.[1] These facts have, more-

[1] It is to be feared that the flow will be greater with chestnut plantations under fruit with the brushwood carefully cleared away but open than on ground covered with dense thickets without chestnut-trees.

over, been verified as regards the large rivers of France : the Rhone, the Loire, the Rhine, the most disastrous floods on which have been attributed to the felling of the trees on the mountains.[1] It would be unfortunate if the people of Corsica, already so tried by the economical crisis, which presses more heavily on that country than elsewhere, had also to undergo, in their turn, this sad trial, the immediate consequence of which would be the abandonment of the low grounds formed by the alluvium of the rivers ; these soils being the most fertile and the only ones whose cultivation is still remunerative. There is an equally important point of view, which it is permissible to glance at, that is the possibility of Corsica being left to its own resources alone. Quite a recent experience, a simple strike of porters,[2] risked placing the people at their last resource. What would happen in time of war if this country were deprived for long months of outside imports ? We are told that the War Department, as a precautionary measure, takes care to provide stores. Would it not be preferable to preserve to Corsica its own crops, like the chestnut-trees, which are at the door of every family and the fruits of which can, in grave circumstances, suffice for the primary needs of the people ? No one can contest the gravity of this felling. It is true that in certain localities they have rested content with thinning the chestnut plantations, and that, on the other hand, the stocks still remain in the ground, but the bare tracts are already numerous, their area increases from day to day, and the consecutive misery of bad harvests aiding the contractors, it is easy to foresee the date when this deafforestation will be complete. The two factories which extract gallic acid[3] (or chestnut extract for tannery purposes) from chestnut-tree wood and which at present exist in the arrondissement of Bastia, absorb 140 tons of wood a day, corresponding to thirty or forty plants, say almost a hectare of chestnut-trees. To that must be added the wood which is exported and that which will be absorbed by a third factory in process of construction, and it will be seen that in less than fifty years the chestnut-trees of Corsica will have succumbed. The Agricultural Societies and the General Council are troubled at this state of affairs, and have asked for exceptional measures for dealing with those proprietors who allow their plantations to be destroyed ; but these solicitations struck against the rights of property and have had no result. It is urgent, however, to find a remedy for this state of affairs,

[1] The inundations of the 16th and 18th October, after the Congress, and produced not by the overflow of the rivers but by torrents from deafforested tracts, have shown how well founded were our apprehensions.—F. D.

[2] The Marseilles strike.

[3] Since 1901 two new factories are at work.

whilst the interest of the proprietors and that of the public are at the same time reconciled. The chestnut woods on the hill slopes may be divided into two categories, those which have been felled and which are transformed into thickets, and those which are still intact. Nothing would be more simple than to cause the first to benefit by the law of 1860,[1] which renders planting obligatory on slopes and the summits of the mountains, exempting them from all taxes for thirty years.[2] This advantage would induce the proprietors themselves to solicit the transfer of their thickets to the forest regulations and to reconstitute their chestnut woods. They would preserve all their rights on the new plantations, except felling, which would be done under the control of the Forest Department. In regard to chestnut woods still in existence or in process of exploitation, and which it would be desirable to preserve, it would be advisable that these also should be put under the management of the forestry department, at the same time, we repeat, paying due respect to the rights of the proprietor. At the present day the contractors cut all the trees; all fall under the blind axe of the butcher, the youngest trunks as well as the very oldest, which they leave in great part on the ground as unsuitable for their industry, and these lands are afterwards given over to the inroads of cattle.

"The function of the forest agents would be to single out the trees to be preserved amongst the youngest and the very oldest, so as to render felling less damaging, from a foresting point of view and to assure the replanting of the felled portions. In exchange for this right of control, the proprietors would benefit by exemption from taxes for thirty years. At the expiry of that time the State would still maintain its right, and the proprietors could only fell or make a clearance of their chestnut plantations in so far as the interests of arboriculture demanded. These measures are of an administrative nature. It will suffice to indicate them. There are others of an arboricultural order, which devolve more specially upon our functions, and the effect of which would be to oppose deafforestation on the slopes of the mountains by rendering the growth of the chestnut more profitable. It is a known fact that deafforestation has commenced, and is being actively pursued chiefly in localities where the cost of harvesting is the greatest, either owing to the poor quality of the chestnuts and their low price, or owing to want of labour. In those districts where chestnuts are sold fresh, and where they yield good quality flour, proprietors do not rob themselves of their chestnut-trees so readily, and contractors do little business there. If it were, therefore, possible to diminish the cost price whilst im-

[1] Repealed by the law of 4th April, 1882. [2] Art. 226 du Code Forestier.

proving the quality of the product, it is certain that proprietors would forego their trees less readily. That is the problem which we have tried to solve, by modifying the system of drying in use which is a most defective one."

Confining ourselves to the subject of the chestnut only, it will be unnecessary to follow the author of this important communication further, but it may be added that it fully confirms the present situation of the chestnut plantations in France, and with an Inspector of Rivers and Forests,[1] an authority on the subject, the authors enunciate the following thesis: *The chestnut-tree will disappear from France if the tannic acid extract industry continues to fire upon it with red-hot bullets.* This disappearance of the chestnut will be a great misfortune from a local point of view, regard being had to the food of the peasants, and a disaster from a general point of view, by still further increasing the denuded surface of the mountains. Moreover, the disappearance of the chestnut will ruin the chestnut extract industry. There exist in France a considerable amount of coppice lands—individual and communal[2]—planted with oak and exploited at short intervals, the revenue of which falls to nothing with the extremely low price of barks. These coppices, thinned at twenty years, reserving 300 standards per hectare (120 per acre), can at the next interval provide 300 oaks forty years old capable of yielding 50 tons of twisted trunks unfit for working, as the limestone of the south cannot elongate the stock of the oak, but, having grown bathed in sunlight, they are capable of supplying the extract factories.

On that account it would be advisable to substitute oak extract for chestnut extract. Now it is known that in tanneries, except for tanning sole leather (*lisse*), a well-manufactured oak extract would have, on the initiation of the Hungarian factories, a big outlet sufficient from the very beginning to keep several extract factories at work, who would then find themselves provided with oakwood, indefinitely, by applying the system of replanting indicated above. On the other hand, the tables prepared by Mangin in his tabulation of the statistics of 1902, show what is the actual quantity of chestnut-trees in France, *viz.*, 90,000 hectares (225,000 acres) in chestnut coppices, and 350,000 hectares (875,000 acres) in chestnut woods. If it be taken that on an average fifty chestnut trees represent 1 hectare of a chestnut plantation and that the average weight of a tree when exploited is about 2 tons, the total amount of chestnut trees in France will be represented by the formula: 350,000 hectares with 50 trees = 17,500,000 trees, or in kilogrammes, 17,500,000 trees at 2,000 kilogrammes (say

[1] M. Teissier. [2] About 2,800,000 hectares (say 7,000,000 acres).

2 metric tons) = 35,000,000,000 kilogrammes (say 35,000,000 metric tons). As the French extract factories (see Chapter IV.) consume annually 450,000,000 kilogrammes (say 450,000 metric tons) (certain factories work Sundays and holidays (*fêtes*), so it is necessary to calculate 500,000 tons as the annual consumption), the actual stock, which has just been estimated, will therefore be exhausted in 35,000,000 ÷ 500,000 = 70 years—that is supposing that this amount was available. Now a number of chestnut-trees are preserved as fruit-trees, others for some other reason, and finally others grow in spots where they cannot be got at; this quantity, which may be called a reserve factor, is difficult to estimate and disturbs the calculation of the longevity of the present factories. It would be easy to ensure this longevity by a little replanting, because it suffices, from the above figures, to replant fifty trees per hectare (twenty trees per acre) per annum; each tree grows 0·050 cubic metres annually (say 1,500 kilogrammes per hectare, say 600 kilogrammes or 1,320 lb. per acre); the 350,000 hectares (875,000 acres) could easily produce, without being impoverished, 525,000 tons per annum. To conclude, therefore, to maintain the present prosperity of extract factories, it will suffice if each large chestnut-tree felled be replaced by two young plants—that is an affair of 400,000 plants annually, which the State or departments may distribute gratuitously; an easy matter to organise. Finally, according to other estimations (on the Savoys) it will be necessary to allow for an annual average production of 4·6 cubic metres per hectare, say more than 900,000 tons per annum. In any case the remedy of the felling of the chestnut plantations is easily carried out, if regard be had to the preceding and if prompt and thorough action is taken against this deplorable state of affairs.

Yield of Chestnut Wood.—The yield of chestnut wood in extract of 25° evidently varies with the nature of the tree treated. It is also a factor of its hydrogenated condition, of the district or place in which it has grown.[1] The wood of the South of France is richer than that of the North or even of Central France, likewise those of the same district but grown with a southern aspect are richer than those with a northern exposure. If it were practically possible to select the trees according to these principles the tannin industry would gain much; unfortunately with the present felling it is not possible even to dream of it. It is necessary to take things as they come. The average yield of a well-installed and improved factory, in which the manufacture is controlled may be given as taking 450 kilogrammes of chestnut wood with 40

[1] Fliche and Grandeau, "On the Influence of the Chemical Composition of the Soil on the Growth of the Chestnut," *Ann. de Chim. et de Phys.*, 5e Series, vol. iii., 1876.

per cent. moisture, that is to say after a year in the shed, or 550 kilogrammes with 55 to 60 per cent. moisture, to produce 100 kilogrammes of extract at 25° B. This yield may be commercially assured, and that whether intensive working be adopted, that is to say, to obtain from a given installation a maximum production, or whether rational working be pursued, which consists in forcing the maximum yield, by first drawing off the quintessence of the juice contained in the decantation mud (by the authors' processes described farther on) and by a more methodical extraction of the wood in autoclaves or in the open air. In principle, the authors recommend this rational method because it realises greater profit to that yielded by the intensive method, which leaves too much to be desired, from an extraction point of view, and the percentage of tannic acid in the product. Several factories have already adopted the rational method, which, although producing less than the intensive method with equal capacity of plant, yet yields a very liberal compensation by the quality and strength of the extract obtained, whilst still maintaining the same yield.

TABLE II.—SHOWING THE DIFFERENT YIELDS OF CHESTNUT-TREES BY THE RATIONAL METHOD OF TANNIN EXTRACTION

50 per cent. Lyonnais. „ „ Dauphiné.	Dry Branches.	Green Branches.	Dry Roots.	Fresh Roots.	Roots and Branches.	Stripped Wood.	Corsican Stripped Wood.	Gard Stripped Wood.[1]
	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.
Yield of wood in 20° extract per cent. .	31·7	31·5	39·0	42·0	35·9	35·0	44·0	32·5
Yield of wood in 25° extract per cent. .	24·8	24·0	31·0	33·0	28·0	28·0	35·0	26·0

All these tests were made on a minimum of 20 tons of wood or roots, and using maceration by seven washings and recuperation of the mud, according to the authors' system. The pressure in boiling was uniform in all the tests, that is to say 1·5 kilogrammes maximum for all the boilings. The strength of the liquors oscillated from 4·3 to 5° B.

The Influence of the Water used for Diffusion of Wood and Bark.—The importance of this question being granted, and seeing the authoritative work on the subject by Nihoul, director of the Tanning School of Liège, on the influence of the water used in the maceration or the extraction of tannin from wood or bark in the tannin industry or in tanneries, the authors do not hesitate to publish it *in extenso*, the more so as it plainly confirms the industrial results obtained by the authors in this special tannin industry.

[1] 40 per cent. moisture.

Note on the Transformations which are Produced in the Infusion of Tanning Materials. By Ed. Nihoul and L. van de Putte.[1]—In a previous research[2] we have shown that the loss in tannin produced during the extraction of tanning materials by saline solutions was most often independent of the increase in insoluble matter. In other words, it is not owing to the formation of insoluble tannates that there is a diminution of assimilable matter by skin powder in the conditions incidental to the analysis of tannins. We found that these losses arise, partially or wholly, from the transformation of the tannin into non-tannin, the latter remaining in solution in the infusion and passing through the filter of powdered skin without being absorbed. In some cases, however, there is manifestly produced a precipitate of a tannate of an alkaline earth which certainly has greatly increased the loss. On the other hand, we have determined that more generally the bright infusions obtained become turbid somewhat rapidly after filtration, even infusions obtained by the use of distilled water. The fact is not new, however, as far as the last point is concerned. Dr. Paessler even attributes to these precipitates, which eventually form in tannin liquors intended for analysis, the errors in the tannin obtained, when the last portions of the liquid which pass through the filter are used for the determination of the total extract.[3] When extracting is done in the factory for the manufacture of the liquor, distilled water is not employed, but the tannery water, generally well water, and consequently charged with mineral matter. There would be nothing impossible, therefore, in that case, if the substances in solution should intervene to modify the solubility of these slightly soluble tannins, to which Paessler attributes the precipitation phenomena, which are produced in the infusions intended for analysis. In the case of hard waters the precipitate is peculiarly abundant, and occurs very rapidly after filtration. We tried to ascertain whether the deposit thus formed was due to matters assimilable by the skin, and, if so, what were the losses of tannin caused by this factor in the liquor. The importance of the question will be conceived, for in that case the phenomenon would result in a still further increase in the loss in tannin mentioned in our previous research. In fact, from the rapidity of precipitation it would seem that the greater part of these insoluble tannins escape the action of the hide, and are deposited at the bottom of the vats

[1] Communication to the Liège Section at its meeting on 12th November, 1903.

[2] E. Nihoul and L. van de Putte, "Effect of the Chlorides and Sulphates of Natural Waters on the Extraction of Tanning Substances," *Bull. Assoc. Bel. des Chimistes*, 1903, Nos. 8, 9 and 10, pp. 298 *et seq.*

[3] *Deutsche Gerber Zeitung*, 1901, No. 132 to 142; *Bull. Assoc. Bel. des Chimistes*, 1904, pp. 115-123.

or on the surface of the leather without penetrating it. The examination of this question led us to wish to ascertain, first of all, what happens when the extraction is done with distilled water, and we have been able to find a much more important cause than that found by our colleague, Dr. Paessler, of the loss in tannin which tannin infusions undergo after being preserved for some time. It is the results of this research which are now given, reserving for future publication our practical tests with tannery waters. Three commercial tanning materials —oak bark, pine bark and sumac—were analysed by the method of the International Association of Leather Chemists and gave the composition embodied in Column I. of the following table :—

TABLE III.—EXPERIMENTS ON THE EXTRACTION OF TANNING MATERIAL USING DISTILLED WATER AS A SOLVENT. OAK BARK

	I. Normal Infusion.	II. Same diluted one-half.	D.	III. Same after three days.	D.	IV. Same plus Thymol.	D.	V. Same Sterilised.	D.
Extract total . .	20·13	19·70	−0·43	18·80	−1·33	20·03	−0·10	19·82	−0·31
„ ash . .	0·89	0·83	−0·06	0·98	+0·09	1·10	+0·21	1·03	+0·14
„ organic .	19·24	18·87	−0·37	17·82	−1·42	18·93	−0·31	18·79	−0·45
Non-tannin total .	5·31	5·17	−0·14	4·52	−0·79	5·43	+0·12	5·96	+0·65
„ ash .	0·76	0·79	+0·03	0·86	+0·10	0·91	+0·15	0·96	+0·20
„ organic	4·55	4·38	−0·17	3·66	−0·89	4·52	−0·03	5	+0·45
Total matter fixed by skin . .	14·82	14·53	−0·20	14·28	−0·54	14·60	−0·22	13·86	−0·96
Mineral matter fixed by skin .	0·13	0·01	−0·09	0·12	−0·01	0·19	+0·06	0·07	−0·06
Tannin . . .	14·69	14·49	−0·20	14·16	−0·53	14·41	−0·28	13·79	−0·90
Water . . .	10·35	...	...	...	...	...	...	...	...
Insoluble . .	69·52	...	...	...	...	...	...	...	...

TABLE IV.—EXPERIMENTS ON PINE BARK

	I. Normal Infusion.	II. Same diluted one-half.	D.	III. Same after three days.	D.	IV. Same plus Thymol.	D.	V. Same Sterilised.	D.
Extract total . .	27·62	26·89	−0·73	25·62	−2·00	25·73	−1·89	26·14	−1·48
„ ash . .	0·35	0·60	+0·25	0·71	+0·36	0·74	+0·39	1·12	+0·77
„ organic .	27·27	26·29	−0·98	24·91	−2·36	24·99	−2·28	25·02	−2·25
Non-tannin total .	4·47	4·69	+0·22	4·34	−0·13	4·09	−0·38	5·28	+0·81
„ ash .	0·20	0·31	+0·11	0·45	+0·25	0·53	+0·33	0·93	+0·73
„ organic	4·27	4·38	0·11	83·89	−0·38	3·56	−0·71	4·35	+0·08
Total matter fixed by skin . .	23·15	22·30	−0·95	21·28	−1·87	21·64	−1·51	20·86	−2·29
Mineral matter fixed by skin .	0·15	0·29	+0·14	0·26	+0·11	0·21	+0·06	0·19	+0·04
Tannin . . .	23·00	21·91	−1·09	21·02	−1·98	21·43	−1·57	20·67	−2·33
Water . . .	12·48	...	...	...	...	...	...	...	...
Insoluble . .	59·90	...	...	...	...	...	...	...	...

TABLE V.—EXPERIMENTS ON SUMAC

	I. Normal Infusion.	II. Same diluted one-half.		III. Same after three days.		IV. Same plus Thymol.		V. Same Sterilised.	
			D.		D.		D.		D.
Extract total . .	35·54	34·83	− 0·71	35·32	− 0·22	35·14	− 0·40	35·25	− 0·29
„ ash . .	2·50	2·47	− 0·03	2·54	+ 0·04	2·63	+ 0·13	2·93	+ 0·43
„ organic .	33·04	32·36	− 0·68	32·78	− 0·26	32·51	− 0·53	32·32	− 0·72
Non-tannin total .	16·63	16·80	+ 0·17	17·91	+ 1·28	17·21	+ 0·58	17·64	+ 1·01
„ ash .	2·46	2·35	− 0·11	2·48	+ 0·02	2·48	+ 0·02	2·66	+ 0·20
„ organic	14·17	14·45	+ 0·28	15·43	+ 1·26	14·73	+ 0·56	14·98	+ 0·81
Total matter fixed by skin . .	18·91	18·03	− 0·88	17·41	− 1·50	17·93	− 0·98	17·61	− 1·30
Mineral matter fixed by skin .	0·04	0·12	+ 0·88	0·06	+ 0·02	0·15	+ 0·11	0·27	+ 0·23
Tannin . . .	18·87	17·91	− 0·96	17·35	− 1·52	17·78	− 1·09	17·34	− 1·53
Water . . .	8·67	...	...	...	...	...	...	...	...
Insoluble . .	55·79	...	...	...	...	...	...	...	...

The tanning substances in these three raw materials were afterwards extracted in Koch's apparatus,[1] using distilled water and working on 27 grammes of oak bark, 17·8 grammes of pine bark, 17·5 grammes of sumac. The last portions of the infusion were concentrated so as to obtain 1 litre of infusion with each substance. The solutions were then filtered so as to obtain a limpid filtrate, and the first 100 c.cm. which passed through was rejected in each case so as to avoid the errors occasioned by the absorption of tannin by the filter paper. From the remainder, after rendering the bulk homogeneous, four portions of 150 c.cm. were drawn and treated as follows: To the first 150 c.cm. of distilled water was added and the mixture analysed; the figures given in Column II. were obtained in that way. To the second 150 c.cm. of distilled water was likewise added, but the mixture was not analysed until three days afterwards. The results are given in Column III. To the third part of the infusion 150 c.cm. of distilled water was likewise added and preserved for three days before being analysed, but a small piece of thymol was added to the flask in the beginning to prevent fermentation; the figures obtained on analysis are given in Column IV. The fourth portion was likewise protected from ferments by sterilisation. The operation was conducted as follows: The liquid was run into a 300 c.cm. graduated flask and 100 c.cm. of distilled water added. The flask was closed by an asbestos plug, previously scorched by passage through a Bunsen burner. The content of the flask was brought to the boiling point on three succeeding days for two to three minutes.

[1] Nihoul (Ed.), "Analysis of Tanning Substances and the Yield Obtained in Tanneries," *Bull. Scien. de l'Assoc. des Écoles spéciales de Liège*, 1903, No. 2 *et seq.*

The third day distilled water was added to make up the bulk to 300 c.cm. (Column V.). The first column gives the results of the analysis under the normal conditions of the International Association, whilst in the others the tannin solutions were diluted by one-half. A peculiar thing was that none of these solutions became turbid after three days' standing, from which it may be inferred that the deposits obtained hitherto are due to slightly soluble tannins, which in the present case are held in solution by the excess of solvent. Let us examine what takes place in the case of each tanning material.

Oak Bark.—The total extract has diminished from Column I. to Column II., and this diminution affects both the organic non-tannin and the tannin, the latter a little more so. This diminution is accentuated in Column III., but here the non-tannin is more especially affected, which shows that chemical changes are produced in the tanning liquor during the three days' standing of the solution. These changes do not occur in the presence of thymol, which would seem to indicate that they were due to micro-organisms; Column IV., in fact, shows barely appreciable losses. But the loss in tannin appears again in Column V., and even exceeds the loss undergone by the liquid left to itself for three days; it is almost 1 per cent.; however, the amount of non-tannin has increased by almost 0·50 per cent. This partial destruction of the tannin to the increase of the non-tannin has been already established by Parker and Procter (*Journ. Soc. Chem. Ind.*, 1895, p. 635); it is due to the action of heat on the tannin solution. It is one of the chief causes which led to the method of extraction of the International Society of Leather Chemists. It is also found that the dissolved mineral matter is increased by allowing the tannic solution to stand in the flask. That increase is really due to the action of the infusion on the glass. The authors observed the same fact in leather analysis.[1]

Pine Bark.—The loss in total extract and in organic extract, in the case of oak bark, is repeated as regards the pine, but with greater intensity, and a peculiar thing occurs in the case of the pine, neither the solution to which thymol has been added (Column IV.) nor the sterilised solution escapes this loss. It would, therefore, be impossible in this case to explain the phenomenon by the intervention of lower organisms. The most appreciable losses in total extract and in organic extract occur in Columns III. and IV., and possibly diastatic action may be invoked to explain them. The presence of diastases in the infusions is possible in all cases, for the greater part of the infusions are obtained at a lower temperature than 50° C. (122° F.). The action of the heat

[1] "Chemical Examination of Leather," *Revue Universelle des Mines*, etc., 1901-2, t. lvi.

necessary for sterilisation has as before occasioned a loss in tannin to the profit of the non-tannin. As to the increase in the mineral matter, it is again found in the pine bark as it was in the oak bark.

Sumac.—This tannin shows a more passive behaviour than the preceding. In fact, the decrease in total extract and in organic extract is barely appreciable. It is under the action of heat (Column V., sterilised solution) that the product shows itself the most sensitive as regards organic extract. Here, as in the cases of oak and pine barks, the losses in tannin of the solutions to which thymol was added are less than those of similar solutions, the compositions of which are tabulated in Columns III. and V. As to the mineral matter, here it again increases and agrees with the duration of the contact with the glass flask and the temperature which the infusion had undergone. The general conclusions to be drawn from the experiments may be enunciated as follows :—

(1) Dilution of the infusions intended for analysis injuriously affects the accuracy of the results, the total extract and the total organic extract being lower in dilute solutions. If Columns I. and II. be compared it will be seen that the loss in organic extract especially affects the tannin. It follows that the clause proposed by Professor Procter at the Paris Congress is perfectly justified. So that the results obtained by different chemists may agree, it is indispensable that they operate on infusions the tannin content of which is appreciably the same. Moreover, in two cases out of three it is only the tannin that is affected by that fact, the loss in organic extract being, even as regards the pine and the sumac, slightly lower than the loss in tannin. One might perhaps see in this fact *a direct action of water on the tanning substance*, which might be more easily entrained by the steam during evaporation, as actually occurs with non-volatile organic matter, such as sugar and glycerine. Another influence may very probably intervene in these losses of tannin, the duration of filtration of the original infusion. Paessler found (*loc. cit.*), in fact, that in the filtration of tannin infusions the tannin determined in the filtrate after 400 to 500 c.cm. had run through was slightly less, owing to the precipitation of slightly soluble tannin. The loss sustained under this head, determined on the whole extract, is comparatively small and does not reach more than 0·3 per cent. It even happens that certain tanning substances, like the mangrove, undergo no loss under such conditions. It may again be asked whether the losses so sustained are due to the precipitation of slightly soluble tannins or to the same phenomena indicated in preceding researches. In any case, the differences on the losses in tannin, shown in Column II. on the one hand and in Columns III. and IV. on the other hand, show

that precipitation can only intervene to a slight extent in the losses found.

(2) Those precipitates which are sometimes got in infusions when working by the rules of the International Association, which are dissolved on heating and are eventually reprecipitated on cooling, remain in solution when the amount of solvent is increased.

(3) A third conclusion to be drawn from these experiments is the necessity of immediately submitting tannin infusions to analysis, if it be desired to avoid errors in the tannin determination. Analogous facts have just been observed by Paessler on juice got from myrobolam extract.[1] The explanation of this may be perhaps found in the fact that a tannic acid extract analysed at different intervals often yields slightly different results.

(4) Care must be taken not to heat too strongly during extraction. In our opinion it would be well if the International Association made the rules in this connection more precise. The foregoing considerations may be useful after the slight differences which have occurred in the German[2] section of the International Association regarding the analysis of tannic acid extracts.

Action of Saline Matter on Tannin Infusions and Extracts.—The great importance to the tanner and to the manufacturer of tannic acid extracts of using only the purest possible water containing the smallest amount of saline matter has already been described.[3] For some years back the use of extracts for tanning has become so general in Belgium that several firms use such products alone, and have given up completely the use of oak bark and even of tropical tannins. It will not, therefore, be uninteresting to try to find what may be the effect of tannery waters on solutions of tanning materials, the more so as this question will verify whether osmotic phenomena intervene in the loss of tannin indicated in a preceding research. The formation of more or less abundant precipitates, which form after some time in infusions obtained by the use of hard water, has already been pointed out.[4] The precipitation is more or less abundant and more or less rapid according to the amount of salts held in solution by the water. The following were results obtained with sumac in the first research on this subject.[5] In the experiment with sumac the liquid passed through turbid even when an extra strong filter, Schleicher and Schull's No. 605, was used. It was not until after

[1] "Ueber das Verhalten von Myrobolanen-extracten bei der Aufbewahrung unter Verschiedenen Verhältnissen," *Deutsche Gerber Zeitung.*

[2] *Collegium*, 1903, Nos. 70, 80, 81 and 82.

[3] *Bull. Assoc. Bel. des Chimistes*, 1903, Nos. 8, 9, 10.

[4] *Idem*, 1903, No. 11. [5] *Collegium*, 1902, p. 89.

100 c.cm. of liquid had run through that the liquid was fit to be used or the estimation of the non-tannin. Such phenomena are accentuated in the case of the waters V. and VI. With sumac, filtration was extremely difficult, and it was found that the liquid obtained bright by being passed through the extra strong filter No. 605, immediately became turbid to such an extent that it was necessary to refilter it through a second filter, through which it was passed twice. This fact explains the decrease in the total extract obtained by analysis. The tannery waters referred to gave a fixed residue of 842 milligrammes per litre for the first, 1,843 and 2,070·5 milligrammes for the two others, *i.e.*, waters V. and VI. The losses on ignition, not including the carbonic acid, were in these cases only 76 and 37·5 milligrammes; the fixed residue therefore consisted almost entirely of mineral matter. The lime estimation gave respectively 276·4 and 458 milligrammes, the chlorine 395·5 milligrammes and 465·5 milligrammes, the sulphuric acid 270 and 338·3 milligrammes; the two waters both contained large amounts of calcium chloride. The precipitate formed in sumac infusions, obtained by means of these waters, was so abundant as to cause the organic extract to fall from 33·96 to 18·42 and the tannin from 23·02 to 2·11, evidently disastrous results for the tanner and which astonished the investigator himself. In his researches on the effect of chlorides and sulphates, the reappearance of these precipitates was pointed out, which, owing to the small content of the solutions, were only formed after some time, but, however, quick enough that when these deposits consist of tanning material, they almost completely escape from the tanning, since they are deposited in the bottom of the baths or on the surface of the leather. Besides, it is necessary to ascertain whether chemical reactions cannot after a certain time take place between the tanning material and the salts present, so as to lower the percentage of matter assimilable by skin powder. The following experiments were limited to testing the action of calcium chloride, sodium sulphate and bicarbonate of magnesia. Accordingly infusions of tanning materials were prepared in distilled water to a concentration double that of the normal concentration, that is to say of the concentration prescribed by the International Association of Leather Chemists. As previously the experiments were on oak and pine bark and on sumac. The litre of infusion was in each case divided into 250 c.cm. To the first portion 250 c.cm. of distilled water was added, to the second an equal volume of water containing 0·1 per cent. of calcium chloride, to the third the same volume of a 0·1 per cent. solution of sodium sulphate, to the fourth also an equal volume of a 0·1 per cent. solution of bicarbonate of magnesia. The first vessel therefore contained an infusion in distilled water brought to the normal

degree of concentration, the second a normal infusion in water containing 0·05 per cent. of calcium chloride, the third likewise in water containing 0·05 per cent. of sulphate of soda, and finally in the fourth the tanning material was present in a solution containing magnesium bicarbonate in the proportion of 0·05 per cent. The liquids, thus brought to the right degree of concentration required for analysis, were left in closed flasks exposed to the light for three days in presence of thymol, then filtered and analysed by the method of the International Association of Leather Chemists. The same experiments were repeated on a certain number of commercial tannic acid extracts. Quebracho extract, chestnut extract, and mimosa extract D were tested in this way. Columns I. of the following tables show the results of the analysis of the normal infusion; Columns II. correspond to the calcium chloride solutions; Columns III. to the sulphate of soda, and Columns IV. to the bicarbonate of magnesia. Among the three saline matters used two of them lose weight on ignition; it follows that the mineral matters found in the ash, whether of the total extract or of the non-tannin, will be less than those which go to increase the percentage of total extract and of non-tannin. It is thus, for example, that in the total extract the chloride of calcium is recovered with its six molecules of water of crystallisation, whilst it is anhydrous calcium chloride which is found in the ash of that extract. This would lead in the ordinary calculation of the results to a fictitious increase in the organic extract. It must not, however, be forgotten that this cause of error reappears in the determination of the non-tannin and its ash. That is why it was not taken into account in our previous researches. Sodium sulphate loses its water of crystallisation at 100° C. (212° F.). Now as the total extract and the non-tannin are dried at 105° C. (221° F.), this salt ought to be recovered integrally in the ash. As to magnesium bicarbonate, it apparently should be converted completely into neutral carbonate by evaporation of its solution and into oxide by calcination. It was, however, considered advisable to make a prior test of saline solutions of equal concentration, but not mixed with tannic infusions: 500 c.cm. of these solutions evaporated to dryness, then calcined, gave results the average of which are given in the following table:—

TABLE VI.—SHOWING RELATION OF RESIDUE ON EVAPORATION TO RESIDUE ON IGNITION OF VARIOUS SALTS

	$CaCl_2$ aq.	Na_2SO_4 aq.	$Mg(HCO_3)^2$.
Fixed residue at 105° C. . .	0·365 gramme	0·260 gramme	0·253 gramme
Ash.	0·244 „	0·2593 „	0·114 „

Complete drying was finished in a hot-air oven at 105° C. (221° F.) until of constant weight, and the ignition was continued for the same length of time as during the determination of ash in the analysis of tannic liquors. So as to be able to compare results more easily, the saline matter in solution has been deducted from the weights yielded by the total extract and the total non-tannin. As regards the ash, the result obtained is followed by the result calculated from the preceding data, and from the ash obtained from normal infusions in distilled water, *i.e.*, the figures given under the heading "Ash (calculated)" were obtained by adding to the ash given in the preceding Table VI. that of the ash of the tanning material tested. The organic extract was obtained by deducting from the total extract first the figures corresponding to the fixed residue of the preceding table, and then the ash of the total extract obtained by diffusion in distilled water. The ash (obtained as just indicated) was not utilised (1) on account of the differences which exist between the fixed residue and the ash of these saline matters, and (2) because such ash is subject to slight variation, *e.g.*, calcium chloride, on prolonged ignition at a high temperature, contains quicklime, and the proportion of quicklime is greater the more rapid the ignition. Losses in weight result, which, according to the percentage and the composition of the tanning material present, may sometimes become sufficiently appreciable as to affect to a certain extent the accuracy of the analytical results. If, again, ignition be not prolonged far enough, as calcium chloride undergoes, when heated, first aqueous and then igneous fusion, it may happen that the carbon is imprisoned in the fused igneous mass and then only burns with great difficulty. Errors of a converse nature to the foregoing may thus occur. If these two errors occur in the same analysis, one in the ash of the total extract and the other in the ash of the non-tannin, the analytical results may thus be very greatly affected. Sodium sulphate may also cause errors. As a general rule, it fuses before the complete combustion of the carbon, and a portion of this cannot be burnt, hence a high result. On the other hand it may, especially when heated rather long at a high temperature, be reduced more or less completely by this carbon, without taking into account that other chemical reactions may occur with the ash of the tanning material present. The following experiments were made on this point: (*a*) about ½ litre of sodium sulphate solution at 0·5 gramme per litre was evaporated, the residue on ignition gave as ash 0·2584 gramme; (*b*) the ashes were moistened with 50 c.cm. of pine infusion. After evaporation and ignition 0·2536 gramme was obtained instead of $0·2584 + 0·0027 = 0·2611$; (*c*) the last ashes were moistened, in their turn, with 50 c.cm. of oak infusion. After evaporation

and ignition 0·2572 gramme was obtained instead of 0·2611 gramme + 0·0083 gramme = 0·2694 gramme; (*d*) this freshly ignited residue was moistened with 50 c.cm. of sumac infusion and 0·2778 gramme obtained instead of 0·2694 gramme + 0·0152 gramme = 0·2846. That is to say the ashes due to the sulphate of soda fall, after the action of pine infusion, to 0·2509 gramme; after the action of oak infusion to 0·2462; after the action of sumac they rise to 0·2516 gramme. It is to be observed that in actual analysis these ashes would descend below these figures, because the sodic sulphate operated on would be ten times less. As to bicarbonate of magnesia, it yields oxide by ignition, but the expulsion of the carbonic acid is never complete; moreover, it seems that the bicarbonate is not completely converted into carbonate in the conditions of the determination of the total extract and of the non-tannin, the organic matter present partially hinders its complete decomposition at the temperature of 105° C. (221° F.). Moreover, it will be seen that the actual and the calculated ashes in the tables are seldom represented by the same figures. Endeavours were made to avoid all source of error, because the losses in tannin in the present experiments are far from being so high as in the researches on the influence of saline matter in the extraction of tannins.

First Series of Experiments. Action of Salts on Infusion of Tanning Materials. Oak Bark (Table VII.).—The results of the experiments show that salts of lime and magnesia appreciably altered the percentage of total extract as well as that of the organic extract. The turbidity formed was therefore more abundant in infusions charged with such saline matter. All the liquids had, however, precipitated by the end of the third day, but it seems that in the case of the distilled water solutions and the sulphate of soda solutions, the precipitation was due simply to physical causes, whilst in the case of the two other salts it was rather a chemical cause which occasioned the loss in extract. The non-tannins, total and organic, behave in quite another way, so that calcium chloride seems not to have affected them, and it follows that the loss in tannin is due solely in this case to precipitation. Sodium sulphate, whilst causing a slight loss in tannin, induced a proportional increase in the non-tannin, so that here the loss in tannin is of a chemical nature, the effect of the salt being to change a portion of the tannin into soluble non-tannin. As to magnesium bicarbonate, it caused a loss in organic extract, which was distributed between the tannin and the non-tannin, the last of the two being especially affected by the saline matter.

TABLE VII.—RESULTS OF EXPERIMENTS ON OAK BARK (PER LITRE OF INFUSION)

	I. Normal Infusion.	II. Same containing 0·5 p. 1,000 of $CaCl_2$.		III. Same containing 0·5 p. 1,000 of Na_2SO_4.		IV. Same containing 0·5 p. 1,000 of $Mg(HCO_3)_2$.	
Extract total [1]	4·988	4·760	−0·228	4·950	−0·038	4·834	−0·154
„ ash [2]	0·166	0·582	+0·416	0·706	+0·540	0·386	+0·220
„ „ [3]	0·166	0·654	+0·488	0·686	+0·520	0·394	+0·228
„ actual organic	4·822	4·594	−0·228	4·784	−0·038	4·668	−0·152
Non-tannin total [1]	1·630	1·618	−0·012	1·750	+0·120	1·528	−0·102
„ ash [2]	0·126	0·656	+0·530	0·678	+0·552	0·324	+0·158
„ „ [3]	0·126	0·614	+0·488	0·646	+0·520	0·354	+0·228
„ actual organic	1·504	1·492	−0·012	1·624	+0·120	1·402	−0·102
Fixed by skin total	3·358	3·142	−0·216	3·200	−0·158	3·306	−0·052
Tannin	3·318	3·102	−0·216	3·160	−0·158	3·266	−0·052
„ lost per cent.	0·0	6·51		4·79		1·56	

TABLE VIII.—RESULTS OF EXPERIMENTS ON PINE BARK (PER LITRE OF INFUSION)

	I. Normal Infusion.	II. Same containing 0·5 p. 1,000 of $CaCl_2$.		III. Same containing 0·5 p. 1,000 of Na_2SO_4.		IV. Same containing 0·5 p. 1,000 of $Mg(HCO_3)_2$.	
Extract total [1]	4·212	3·936	−0·276	4·234	+0·022	3·970	−0·242
„ ash [2]	0·054	0·372	+0·318	0·522	+0·468	0·370	+0·316
„ „ [3]	0·054	0·542	+0·488	0·574	+0·520	0·282	+0·228
„ actual organic	4·158	3·882	−0·276	4·180	+0·022	3·916	−0·242
Non-tannin total [1]	0·790	0·916	+0·126	0·940	+0·150	0·958	+0·168
„ ash [2]	0·038	0·648	+0·610	0·586	+0·548	0·232	+0·194
„ „ [3]	0·038	0·526	+0·488	0·558	+0·520	0·266	+0·228
„ actual organic	0·752	0·878	+0·126	0·902	+0·150	0·904	+0·152
Fixed by skin	3·422	3·020	−0·402	3·294	−0·128	3·012	−0·410
Tannin	3·406	3·004	−0·402	3·278	−0·128	3·012	−0·394
„ lost per cent.	0·0	11·80		3·90		11·56	

TABLE IX.—RESULTS OF EXPERIMENTS ON SUMAC (PER LITRE OF INFUSION)

	I. Normal Infusion.	II. Same containing 0·5 p. 1,000 of $CaCl_2$.		III. Same containing 0·5 p. 1,000 of Na_2SO_4.		IV. Same containing 0·5 p. 1,000 of $Mg(HCO_3)_2$.	
Extract total [1]	5·868	5·888	+0·020	5·996	+0·128	5·878	+0·010
„ ash [2]	0·304	0·566	+0·262	0·862	+0·558	0·582	+0·278
„ „ [3]	0·304	0·792	+0·488	0·824	+0·520	0·532	+0·228
„ actual organic	5·564	5·584	+0·020	5·692	+0·128	5·574	+0·010
Non-tannin total [1]	2·654	2·610	−0·044	2·836	+0·182	2·762	+0·108
„ ash [2]	0·354	0·744	+0·390	0·886	+0·532	0·508	+0·154
„ „ [3]	0·354	0·842	+0·488	0·874	+0·520	0·582	+0·228
„ actual organic	2·300	2·256	−0·044	2·482	+0·182	2·408	+0·108
Fixed by skin	3·214	3·278	+0·064	3·160	−0·054	3·116	−0·098
Tannin	3·264	3·308	+0·044	3·210	−0·054	3·166	−0·098
„ lost per cent.	0·00	0·00		1·65		3·00	

[1] Saline matter deducted. [2] Saline matter included. [3] Calculated.

Action on Pine Bark (Table VIII.).—As in the case of oak, the total extract and the organic extract were lowered in the infusions to which calcium chloride and magnesium bicarbonate were added. These salts caused very appreciable loss in tannin. In both cases, besides precipitation of tanning matter, here and there was partial conversion of tannin into soluble non-tannin, as the increase of organic non-tannin in Columns II. and IV. shows. There was therefore partial dissociation of the tanning matter or reaction of the tannin with the saline matter, with production of compounds, non-assimilable by the skin, in the conditions of analysis. Oak tannin behaves in no way the same, because the non-tannins in Columns II. and IV. are, especially in IV., in a decreasing ratio. Oak tannin appears, therefore, less sensitive to these changes than pine tannin. Sodium sulphate behaves similarly as with oak bark, a slight increase in non-tannin with a proportional decrease in tannin. The turbidity formed in the saline infusion was less than that formed in the distilled water infusion, because the organic extract and total extract are slightly increased. Here again the chemical rôle of sodium sulphate in the transformation of tannin into non-tannin is confirmed.

Action of Sumac (Table IX.).—The precipitation was about the same in the four tests. But in the sodium sulphate infusion it was less pronounced than in the other liquids, even than in the distilled water infusion; it follows that the total extract and organic extract only differ slightly. Calcium chloride seems to have no effect on sumac solutions. Sodium sulphate produced a comparatively small loss in tannin, but caused a threefold greater increase in non-tannin, from which one would be inclined to conclude that the slight solvent action of sodium sulphate on the precipitate formed by distilled water acts more on the non-tannin than on the tannin, the more so as the same occurs with pine extract, though to a less extent (Table VIII.). The action of magnesium bicarbonate is more apparent, and the saline matter, without inducing increased turbidity, gives a precipitate of tanning matter or of one or other of its constituents, hence a loss of tannin equivalent to the increase in organic non-tannin. It therefore appears to behave with sumac in a purely chemical fashion in changing tannin into soluble non-tannin.

Conclusion I.—It follows from preceding experiments that saline matters may behave differently towards tanning infusions; they either (*a*) only act slightly, *e.g.*, towards sumac, or (*b*) they induce a loss in tannin without increasing the turbidity, which always occurs in such infusions, even when distilled water has been used in extraction, *e.g.*, action of sulphate of soda on oak and pine infusions. This action of

sulphate of soda is difficult to explain with any degree of certainty. The loss found may be due to a partial change of tannin into a substance unassimilable by the skin, as well as to combination of the tanning matter with the saline matter, so as to form a compound which would not be retained by the skin. It may even be said that sodium sulphate alters the osmotic properties of the skin, so that they are no longer capable of retaining the whole of the tannin. But contrary to this point of view, in no case was any trace of tannin to be found in the liquid passing through Procter's filter. Again, as will be seen farther on, sodium sulphate behaves in quite a different way with concentrated solutions of quebracho extract, in which it induces no loss in tannin. (*c*) They induce a loss in tannin whilst increasing the precipitate formed. This occurs with magnesium and calcium salts, but these can, in certain cases, behave like sodium sulphate, by changing partially the tannin into soluble non-tannin. The turbidity is not always due to a precipitate of tannin, but in certain cases non-tannin is also entrained. It may be said generally that the saline matters, dissolved in natural waters, still further accentuate after extraction the loss already incurred during extraction, whether they precipitate tannin or induce its conversion into bodies unassimilable by skin powder. By the prolonged contact of saline matters with tannic infusions, it may be taken that the losses in tannin become still more appreciable, judging from the precipitates which afterwards occurred in the clear liquors used for analysis. The following are the observations made on these liquors a fortnight after the foregoing tests: (1) The oak solutions were quite turbid except No. 2 (calcium chloride), which showed traces of mycelian growth. (2) The pine solutions remained limpid except No. 2. (3) The sumac solutions likewise remained limpid except No. 4, which presented an abundant deposit. All the infusions containing magnesium bicarbonate were very dark, even sumac infusion.

Conclusion II.—Before drawing the second conclusion which results from our experiments, the losses in tannin which occurred in these latter experiments have to be compared with those found previously during the examination of the action of saline matter on the extraction of tannins. The losses in tannin corresponding to solutions of 0·5 gramme per litre of calcium chloride, sodium sulphate and magnesium bicarbonate were calculated by interpolation from the tabulated results of this latter research, but the results of the present experiments have been brought to 100 parts of the tanning substance used. We thus got the following table, in which are given in horizontal lines corresponding to the letter A, the percentages of tannin when the saline matter

is added to the infusion previously prepared in distilled water, and, in the horizontal lines corresponding to the letter B, the percentages obtained when the extraction is made with the saline solution.

TABLE X.—SHOWING DIFFERENT RESULTS OBTAINED (A) BY EXTRACTING TANNIN BY DISTILLED WATER AND THEN ADDING SALINE SOLUTIONS, AND (B) BY USING THE SALINE SOLUTION FOR EXTRACTION

		I. Distilled Water Infusion.	II. Same with 0·5 p. 1,000 of $CaCl_2$.		III. Same with 0·5 p. 1,000 of Na_2SO_4.		IV. Same with 0·5 p. 1,000 of $Mg(HCO_3)_2$.	
Oak tannin	A.	12·02	11·24	− 0·78	11·56	− 0·46	11·73	− 0·29
	B.		9·04	− 2·98	10·48	− 1·54	8·15	− 3·87
Pine tannin	A.	19·13	17·59	− 1·54	18·63	− 0·50	17·00	− 2·13
	B.		15·10	− 4·03	15·72	− 3·41	16·04	− 3·09
Sumac tannin	A.	18·66	18·69	+ 0·03	18·35	− 0·31	18·10	− 0·56
	B.		16·40	− 2·26	17·12	− 1·54	15·54	− 3·12

The vertical Column I. corresponds, as in the three preceding tables, with results obtained with distilled water; the vertical Column II. corresponds to calcium chloride infusions; Column III. to sulphate of soda, and Column IV. shows the figures obtained in presence of magnesium bicarbonate. The differences between the figures given in the lines A and B are considerable; the losses in tannin observed in the preceding experiments were much lower than those obtained in the experiments relating to the action of salts on extraction. That may be due to two causes: (1) The fact that the absence of vegetable cells containing tanning matters did not induce osmotic phenomena, and consequently the electro-chemical actions to which the previous losses in tannin were attributed. (2) The fact that heat was used during the extraction of the tannins, and that consequently the saline matters were able to act in the hot state more energetically than at the ordinary temperature. It is quite certain that heat intervenes in the results, according to whether the operation is done in the cold or in the hot state. This was well demonstrated in the first examination on the action of Belgian tannery waters in the extraction, but it is especially in the osmotic phenomena that heat intervenes. It is to facilitate this osmosis that the diffusers are heated as much in the sugar industry as in the manufacture of tannin extracts. In the absence of vegetable cells it ought to exert a secondary influence, judging from the results given in the following paragraph, in regard to quebracho extract. That extract was dissolved in the hot state, and consequently the saline matters reacted at a comparatively high temperature. Now the losses in tannin are of the same class as in the preceding experiments. Even sodium sul-

phate has no action any more than in the experiments of Table IX. regarding sumac. There is, therefore, in this comparison of results a confirmation of the explanation given above of the losses in tannin, losses which were attributed to phenomena of an electro-chemical nature. A practical conclusion to draw from that is, that in using a water charged with mineral matters for the extraction of tannin there is every advantage in working on tannin materials ground as finely as possible, if it be desired to prevent the destruction of tannin; it must not be forgotten, however, that the more fine the grinding is the greater is the number of torn cells, and consequently the more is the quotient of purity of the liquor diminished. The influence of this fact is not yet very well understood in tanneries.

Second Series of Experiments. Action of Saline Matter on Tannic Extracts.—A large quantity of tannic extracts being now used in tanning, it is interesting to ascertain what the action of saline water is on the solution of these extracts. The two species of products most commonly used were submitted to the previous tests, *i.e.*, quebracho extract and chestnut extract. Mimosa extract D, made by Le Petit Dollfus and Gansser of Milan, was also tested. It is known that this highly decolorised extract is completely soluble in cold water, and yields quite superior results in tanning. It was tried to ascertain what might be the action of saline matter on this extract on account of the comparatively high amount of ash which it left on ignition, higher even than that of the mineral matter used in the tests.

Quebracho Extract.—The quebracho extract was submitted to three experiments: (1) In the first the amount of material taken was such as to have a concentration double that of the normal concentration prescribed for analysis. The product was dissolved in boiling distilled water, then, after the amount taken for analysis was run into the graduated flask, boiling water was run into it up to three-fourths of its volume, and allowed to cool slowly after adding the saline matter in a volume sufficient to get in the total liquid a concentration of 0·05 per cent. (say 0·5 gramme per litre). Each liquid was analysed on the third day, after filtration and without dilution with an equal volume of water, *i.e.*, in a word, going outside the conditions imposed by the International Association of Leather Chemists. The results obtained figure in Table XI. They show a loss in tannin of 8·6 per cent. as regards the liquor charged with calcium chloride and of 4·8 per cent. as regards the water charged with magnesium bicarbonate. It was observed that the liquid which had passed through the Procter's filter in the first case (Column II.) contained small amounts of tannin, which means that the loss of 8·6 per cent. is exaggerated. Moreover, very peculiarly, it was found that

sodium sulphate, instead of inducing a loss in tannin, on the contrary gave an increase of assimilable matter. These two facts rendered it necessary to make a second series of experiments. (2) This second series was conducted in a slightly different manner. The same quantity of material was weighed and dissolved in, or at least beaten up with, the least possible amount of tepid water, run into the graduated flask, cold water added, and finally the saline matter to be dissolved. The operation in this case was practically done in the cold, whilst in the first case it was done in the hot state. Besides, the filtered liquors after three days were diluted with an equal volume of distilled water before being passed through the skin powder. The results of the analysis are given in Table XII. The loss produced by magnesium salts was about the same, whilst, as anticipated, the loss due to calcic salts had decreased. But the liquid clarified by filtration precipitated at the end of some days. As regards sulphate of soda, curiously enough, it behaved in absolutely the same way as in the first instance. In the two experiments the substances retained by the skin powder were increased by 0·6 per cent. This fact, coinciding with a notable diminution in the ash of the non-tannin compared with the ash of the total extract leads to the conclusion that a portion of the saline matter was absorbed by the skin powder, but as the same fact does not reoccur in Table XII., which gives the results of analysis made on a solution of quebracho extract of normal concentration, it may perhaps be concluded that a possible combination takes place between the saline matters and the tannin of quebracho, a compound which would be absorbed by the hide and which would be produced in presence of an excess of tannin. The high percentages of ash found in leathers tanned with quebracho are perhaps due to similar causes. It appears from a comparison of the two tables that the use of heat to dissolve the extract is prejudicial to the solution of the tannin, the percentage of which approaches, in the second case, that obtained in the normal solution (see Table XIII.). The prolonged action of heat, instead of bringing more matter into solution, induces, on the contrary, in pure water and in water charged with sodium sulphate precipitations which diminish the total extract and the organic extract. But the converse occurs with water charged with salts of the alkaline earths. Another and more important fact, which follows from these experiments, is the increase of the non-tannin induced in these latter liquors by the action of heat. But if solution be effected in the cold, a portion of the non-tannin is precipitated by salts of the alkaline earths. This fact is of importance in regard to the purity of the liquor. (3) The third series of experiments (Table XIII.) was conducted under normal conditions as regards concen-

tration of the solutions. The decrease of total extract and organic extract is in the same ratio as in the preceding series, *i.e.*, the precipitation was less in solutions charged with sodium sulphate and greater in solutions charged with calcium chloride. The percentage of tannin found in the case of a solution of normal concentration approaches that given in the preceding table. In fact, sodium sulphate induces no increase in matter assimilable by the skin ; it, on the contrary, causes a slight loss, of the same nature as that found with infusions of tanning materials.

Chestnut Extract (Table XIV.) is especially sensitive to magnesium bicarbonate, which rather seems to act physically, for it produces no appreciable increase of organic non-tannin. The other saline matters, on the contrary, act chemically, especially sodium sulphate. These salts, however, also act by precipitation, as is shown by the decrease in organic extract. The action of sodium sulphate is quite peculiar, for the like does not occur with any other extract. Moreover, the method of manufacture must in many instances intervene in these changes in composition. The loss caused by magnesium bicarbonate, already so great, would increase with time, because the liquid, clarified by filtration, showed an abundant deposit in a fortnight. The ash of the non-tannin is less by half than that of the total extract. It would, therefore, seem that here a portion of the compound which the tannin forms with the salt was absorbed by the skin powder, so that the actual loss in tannin would really be greater than shown by the figures.

Mimosa D. (Table XV.) is entirely soluble in cold water. It gives no precipitate, either with sodium sulphate or with magnesium bicarbonate. Calcium chloride alone causes turbidity. Independently of this physical action, all the three saline substances always reacted chemically. Sodium sulphate only produced a slight loss in tannin, inducing a proportional increase in non-tannin. Its action is analogous with all the substances examined, chestnut extract excepted. This fact is interesting, because it seems to augur that the ordinary purification of selenitic water may yield good results as far as the solution of tannic extracts is concerned. Magnesium salts, or at least the bicarbonate, have also little effect, but it is not the same with calcic salts, which behave to mimosa like to chestnut extract by reacting physically and chemically. After a fortnight the three clear solutions of mimosa extract were still perfectly limpid. A curious thing is that the mineral matter contained in this extract seems to have no effect of any kind on its composition, whether such mineral matter consists of inert salts, sodium sulphate, for example, or whether it exists as organic compounds. In the latter case they are not, in any eventuality, retained by skin powder, as is shown by the ash of the non-tannin.

Summing up, it may be said that in these experiments the tannic substances examined behave towards saline matter like infusions of tanning substances. The losses are small compared with those produced in the extraction of the latter. Moreover, a little less regularity is noticeable with tannic extracts, but it is possible that the method of manufacture must be taken into account.

TABLE XI.—RESULTS OF FIRST EXPERIMENTS ON QUEBRACHO EXTRACT. DOUBLE NORMAL INFUSION—EXPRESSED AS PER CENTS. BY WEIGHT OF EXTRACT TESTED

	I. Double Normal Infusion.	II. Same with 0·5 p. 1,000 of $CaCl_2$.		III. Same with 0·5 p. 1,000 of Na_2SO_4.		IV. Same with 0·5 p. 1,000 of $Mg(HCO_3)_2$.	
Extract total [1] . .	42·250	41·120	−1·130	42·350	+0·100	41·530	−0·720
„ ash [2] . .	1·325	2·850	+1·525	3·875	+2·550	2·580	+1·255
„ „ [3] . .	1·325	3·765	+2·440	3·925	+2·600	2·465	+1·140
„ actual organic . . .	40·925	39·800	−2·125	41·025	+0·100	40·205	−0·720
Non-tannin total [1] .	5·100	7·200	+2·100	4·575	−0·525	6·190	+1·090
„ ash [2] .	1·425	2·100	+0·675	2·225	+0·800	3·875	+2·450
„ „ [3] .	1·425	3·865	+2·440	4·025	+2·600	2·565	+1·140
„ actual organic . . .	3·675	5·775	+2·100	3·150	−0·525	4·770	+1·095
Fixed by skin . .	37·150	33·920	−3·230	37·775	+0·625	35·340	−1·810
Tannin . . .	37·250	34·025	−3·225	37·875	+0·625	35·435	−1·815
„ loss per cent.	0·0	8·6		0·0		4·86	

TABLE XII.—RESULTS OF FIRST EXPERIMENTS ON QUEBRACHO EXTRACT. DOUBLE NORMAL SOLUTIONS—EXPRESSED IN PER CENTS. BY WEIGHT OF EXTRACT TESTED

	I. Double Normal Infusion.	II. Same with 0·5 p. 1,000 of $CaCl_2$.		III. Same with 0·5 p. 1,000 of Na_2SO_4.		IV. Same with 0·5 p. 1,000 of $Mg(HCO_3)_2$.	
Extract total [1] . . .	44·77	38·30	−6·47	43·50	−1·20	40·29	−3·48
„ ash [2] . . .	1·25	...	...	...	...	...	...
„ „ [3] . . .	1·25	...	...	...	...	...	...
„ actual organic .	43·52	37·05	−6·47	42·25	−1·20	39·04	−3·48
Non-tannin total [1] . .	5·2	1·40	−3·80	3·35	−1·85	2·49	−2·71
„ ash [2] . .	1·05	...	...	...	...	...	...
„ „ [3] . .	1·05	...	...	...	...	...	...
„ actual organic .	4·15	0·35	−3·80	2·30	−1·85	1·44	−2·71
Total fixed by skin . .	39·57	36·90	−2·67	40·15	+0·58	37·80	−1·77
Tannin	39·37	36·70	−2·67	39·95	+0·58	37·60	−1·77
„ loss per cent. . .	0·0	6·78		0·0		4·50	

[1] Saline matter deducted. [2] Saline matter included. [3] Calculated.

TABLE XIII.—RESULTS OF SECOND EXPERIMENTS ON QUEBRACHO EXTRACT. NORMAL—EXPRESSED IN PARTS BY WEIGHT PER CENT. OF EXTRACT TESTED

	I. Normal Infusion.	II. Same with 0·5 p. 1,000 of $CaCl_2$.		III. Same with 0·5 p. 1,000 of Na_2SO_4.		IV. Same with 0·5 p. 1,000 of $Mg(HCO_3)_2$.	
Extract total[1] . . .	45·04	42·50	−2·54	43·90	−1·14	43·19	−1·85
„ ash[2] . . .	2·30	3·50	+1·20	6·25	+3·95	4·20	+1·90
„ „ [3] . . .	2·30	7·18	+4·88	7·50	+5·20	4·58	+2·28
„ actual organic .	42·74	40·20	−2·54	41·60	−1·14	40·89	−1·85
Non-tannin total[1] . .	5·85	4·85	−1·00	5·45	−0·40	6·19	+0·34
„ ash[2] . .	1·90	6·75	+4·85	6·55	+4·65	4·05	+2·15
„ „ [3] . .	1·90	6·78	+4·88	7·10	+5·20	4·18	+2·28
„ actual organic.	3·95	2·95	−1·00	3·55	−0·40	4·79	+0·34
Total fixed by skin . .	39·19	37·65	−1·54	38·45	−0·74	37·00	−2·19
Tannin	38·79	37·25	−1·54	38·05	−0·74	36·60	−2·19
„ loss per cent. . .	0·0	3·97		1·91		5·64	

TABLE XIV.—RESULTS OF EXPERIMENTS ON CHESTNUT EXTRACT—EXPRESSED AS PER CENTS. BY WEIGHT OF EXTRACT TESTED

	I. Normal Infusion.	II. Same with 0·5 p. 1,000 of $CaCl_2$.		III. Same with 0·5 p. 1,000 of Na_2SO_4.		IV. Same with 0·5 p. 1,000 of $Mg(HCO_3)_2$.	
Extract total[1] . .	37·332	35·441	−1·891	35·989	−1·343	34·667	−2·665
„ ash[2] . .	0·803	2·810	+2·007	3·723	+2·920	2·445	+1·642
„ „ [3] . .	0·803	5·683	+4·880	6·003	+5·200	3·083	+2·28
„ actual organic	36·529	34·638	−1·891	35·186	−1·343	33·864	−2·665
Non-tannin total[1] .	9·855	10·402	+0·547	10·804	+0·949	10·030	+0·175
„ ash[2] .	0·839	4·088	+3·249	3·942	+2·103	1·094	+0·255
„ „ [3] .	0·839	5·719	+4·880	6·039	+5·200	3·119	+2·280
„ actual organic . . .	9·016	9·5635	+0·547	9·965	+0·949	9·121	+0·175
Total fixed by skin .	27·477	25·039	−2·438	25·185	−2·292	24·637	−2·840
Tannin . . .	27·513	25·074	−2·439	25·221	−2·292	24·672	−2·841
„ loss per cent. .	0·00	8·86		8·31		10·31	

TABLE XV.—EXPERIMENTS ON MIMOSA EXTRACT D—EXPRESSED IN PER CENTS. BY WEIGHT OF EXTRACT TESTED

	I. Normal Infusion.	II. Same with 0·5 p. 1,000 of $CaCl_2$.		III. Same with 0·5 p. 1,000 of Na_2SO_4.		IV. Same with 0·5 p. 1,000 of $Mg(HCO_3)_2$.	
Extract total[1] . . .	48·44	46·70	−1·74	48·50	+0·06	48·39	−0·05
„ ash[2] . . .	6·44	9·60	+3·16	12·10	+5·66	9·30	+2·86
„ „ [3] . . .	6·44	11·32	+4·88	11·64	+5·20	8·72	+2·28
„ actual organic .	42·00	40·26	−1·74	42·06	+0·06	41·95	−0·05
Non-tannin total . .	10·55	12·50	+1·95	11·40	+0·85	12·19	+1·64
„ ash[2] . .	6·30	11·40	+5·10	12·15	+5·85	8·85	+2·55
„ „ [3] . .	6·30	11·18	+4·88	11·50	+5·20	8·58	+2·28
„ actual organic	4·25	6·20	+1·95	5·10	+0·85	5·89	+1·64
Total fixed by skin . .	37·89	34·20	−3·69	37·10	−0·79	36·20	−1·69
Tannin	27·75	34·06	−3·69	36·96	−0·79	36·06	−1·69
„ loss per cent. .	0·0	9·74		2·08		4·46	

[1] Saline matter deducted. [2] Saline matter included. [3] Calculated.

Various Chestnut Liquors and Extracts. Analytical Tables showing Compositions.—Commercially, it requires on an average 1,300 to 1,400 kilogrammes of liquor of the following composition, which generally titrates 3·8° to 4·5° B., to produce 100 kilogrammes of chestnut extract of 25° B., say a yield of 13 to 14 per cent. :—

TABLE XVI.—SHOWING COMPOSITION OF VARIOUS CHESTNUT WOOD LIQUORS AND EXTRACTS.

	A.	B.	C.	D.	E.	F.	G.	H.
Soluble tanning substances .	3·92	29·20	29·25	28·38	34·30	36·00	19·60	43·00
Non-tannins .	2·36	10·0	13·16	10·69	15·80	14·00	12·20	37·70
Water . .	93·47	60·60	57·42	60·11	48·90	48·70	66·30	19·00
Insoluble . .	0·25	0·20	0·17[1]	0·82[1]	1·00	1·30	1·90	0·30
	100·00	100·00	100·00	100·00	100·00	100·00	100·00	100·00

A, chestnut liquor, total soluble, 6·28.—B, chestnut extract 25°, rational manufacture, from a factory treating a mixture of Lyonnais, Charollais and Dauphiné wood. Dry residue 0·2, density at 18° C. = 1·207, 25·2° B. A well-clarified extract, pretty well decolorised, scarcely becoming turbid when cold, even after long standing of the solution.—C, 25° chestnut extract made at the factory at Génolhac, Gard, by Ausset and Hermet. Very fluid, decolorised, very readily soluble in the cold.—D, 25° chestnut extract from the factory of Roubin et Cie. at Lalevade, Ardèche. Extract well clarified and decolorised, very soluble in the cold, made exclusively with Ardèche wood.—E, 30° chestnut extract of La Société des produits Chimiques de Saint Chamond, Loire. Pretty well decolorised, gives a slight turbidity with cold water and deposits rapidly on standing. This factory works a mixture of Lyonnais, Auvergne and Vivarai wood.—F, analysis of 30° chestnut extract from a Corsican factory. Clarified, but only slightly decolorised, with a somewhat thick deposit on standing.—G, 20° chestnut extract ("Gallique") for dyeing, made as mentioned from unbarked wood and the concentration of the crude liquors from the maceration (under a pressure of 2 kilogrammes). This extract becomes cloudy in cold water and even in hot water, yields a dark brown solution which is, however, of no importance, as this product is intended for dyeing or for the manufacture of boiler fluid.—H, dry chestnut extract made by concentrating 25° extract at 45° in a rotary apparatus *in vacuo*. Very soluble in water, where it only gives a slight turbidity.

[1] Ash.

CHAPTER II

PLANT AND EQUIPMENT FOR TREATING CHESTNUT WOOD

System of Heating—Steam Boilers—Management and Upkeep—Furnaces and Various Gazogenes—Chimneys—Steam Engines—Cutters—Cup Elevators—Conveyers

Boilers with Two "Bouilleurs" with or without Reheaters.—The system, with great reserves of water and steam, with economical results, allowing the fixing of grates of large section for the adaptation of generator (gazogenes) furnaces burning the waste or chips of the exhausted wood is that to be recommended for extract factories. Fig. 64 shows a boiler of this kind.

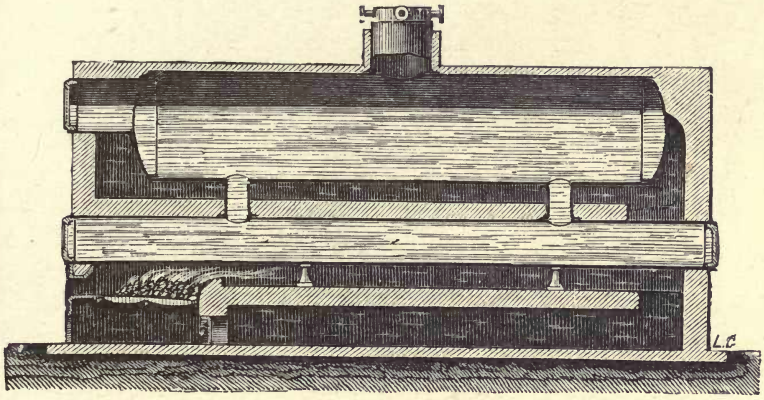

FIG. 64.—Boiler with two *bouilleurs*.

"Multibouilleur" Boilers with Reheaters.—System, with great reserves of water and steam, allowing the use of very variable quantities of steam without inconvenience. Methodical heating, easy cleaning. Very good calorific results. Recommended for large tanneries and extract factories, permitting also the special adaptation of gas generator (gazogenes) furnaces burning spent tan or exhausted wood chips with 55 or 60 per cent. of moisture. Fig. 65 shows a boiler of this kind; the tops of the

heads of the *bouilleurs* and the reheaters are made of stamped steel. In a general way, in normal working the boiler vaporises about 14 kilogrammes per square metre of heating surface.

Boilers with Interior Furnaces and Galloway Tubes.—Finally, a type of this kind is shown in Fig. 66. It is that which is most suitable for an extract factory and in which the calorific utilisation of the hot gases is a maximum, with an enormous evaporating capacity.

Management of the Boilers.—In the case of three boilers, Nos. 1, 2, 3, one of which is standing.

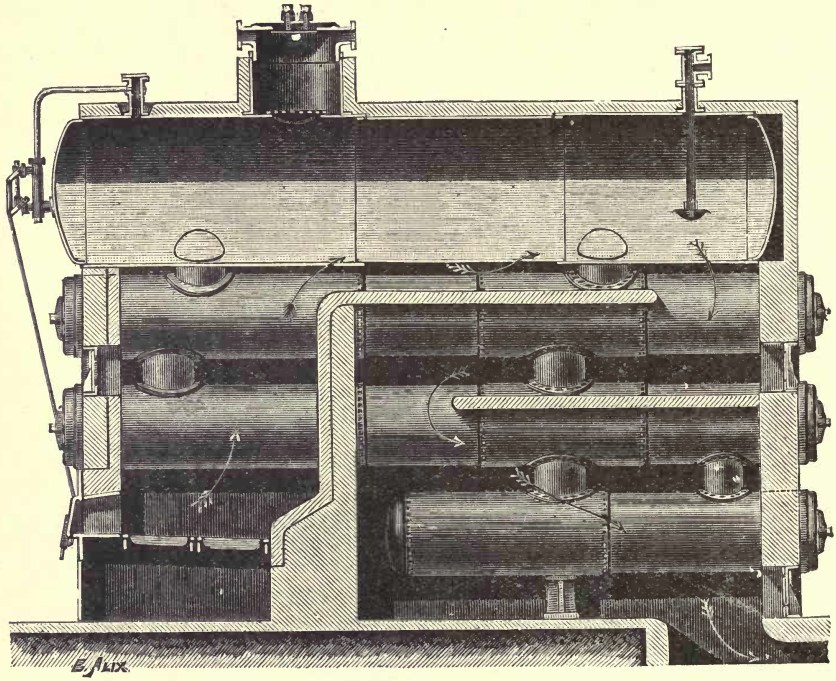

FIG. 65.—*Multibouilleur* boiler.

Rotation of Boilers and Cleaning.—The boiler must be changed every two months, in such a way that each works during four consecutive months. Each time that the boiler is changed, that is to say, every two months in normal working, it is necessary, as soon as the boiler is cold, to empty it, undo the manhole, inspect the inside, scrape off the encrustation if need be, clean the smoke flues, close up the manhole, fill it up again so as to bring it in a state fit for working as soon as possible, so as to cope with any accident to the other boilers.

Extractions.—If the water be strongly calcareous (very hard) it will

be necessary to make an extraction each morning from each boiler so as to remove the mud in the boilers. Each extraction should be about 20 cm. (8 inches) of water.[1] To make an extraction from a boiler all that has to be done is to open the tap of one of the lower *bouilleurs* and allow the level to fall 10 cm. (4 inches), close that tap and open the tap of the other to lower its level 10 cm. (4 inches), and close that tap.

Water-Level.—The water-level ought always to be kept as near as possible to the line marked water-level. It should not be allowed to fall below it, nor rise more than 10 cm. (4 inches) above or below the normal level. If, by accident, the level falls lower, and especially if no water is to be seen in the gauge glass, the boiler is in danger of explosion. In that case it is necessary to feed the boiler immediately and to close the damper completely, so as to stop heating. If the water-level rises higher than 10 cm. (4 inches) above the normal level, there

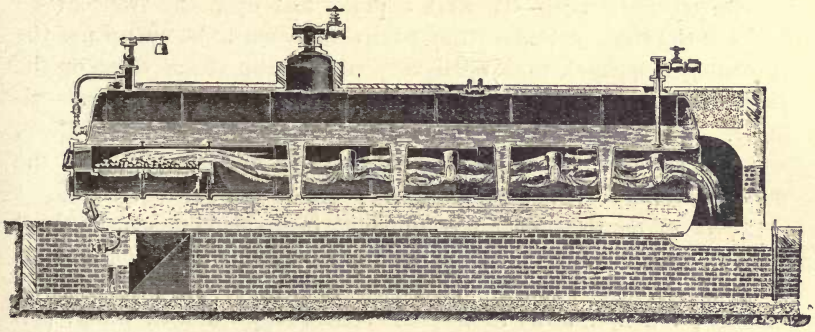

FIG. 66.—Boiler with two interior cylindrical furnaces with Galloway tubes in the furnaces.

is a risk of water being entrained by the steam and of causing the engine cylinders to jump. In that case the water feed must be immediately shut off, and water run off to bring the water to its normal level. These are two of the most grave faults on the part of an engine-driver. They may cause an explosion in the boiler or the steam engines. Each morning the engine-driver ought to manipulate all the water-level (gauge glass) taps, not only those on the cast-iron foundation, but those used for blowing off. This precaution is necessary so that the taps do not become fast and cannot therefore be turned instantly at the moment when a gauge glass breaks.

Pressure.—The driver is entrusted with the manipulation of the registers (dampers) so as to increase or arrest the pressure according to the indications of the pressure gauge. He ought, each morning, to

[1] Noyer's Paratartar prevents encrustation.

mount the boilers, and lift gently and very slightly by hand each of the safety valves, so as to let them blow off. This precaution cleans the seat of the valves and prevents them becoming fast, which may occur when they seldom or almost never blow off. When the pressure rises above the regulated and the valves blow off strongly the principal dampers must be closed, the boilers fed a little more rapidly (unless the water-level be not already too high), and arrangements made with the man in charge of the autoclaves or steam engines to take a little more steam, if possible, for a moment, until the pressure is reduced to its normal figure. The engine-driver ought to repair, forthwith all leakage which occurs in the valves of all the boiler taps.

Feed Pumps and Boiler Valves.—The valve for applying steam, on the feed of the boilers which supplies the feed pumps, ought to be entirely open.

Starting the Pumps.—To start a pump first open the two blow-off cocks of the steam cylinder (they ought moreover to be open since the preceding stoppage), then open very slightly the steam valve on the pump to blow off the condensed water contained in the pipe and cylinder. When no more water issues from the blow-off taps close them, and increase the opening of the steam valve until the speed of the pump is attained.

Speed of the Pump.—One pump alone must be kept constantly at work at reduced speed without ever stopping. This style of working greatly preserves the pumps, and generates more steam from the boilers. When, in order to keep the boilers supplied with water, it is necessary to work a pump at rather high speed, but which never ought to reach more than 80 to 90 strokes, that is a sign that the water piston has much play in its cylinder; it must then be dismantled and its packing renewed.[1]

Stopping the Pump.—To stop the pump the steam valve must be completely closed and the blow-off cocks of the steam cylinder opened.

Pump Joints.—(1) The joints of the bottoms of the cylinders and of the bell for the return air ought to be made from a sheet of thick paper or of thin, carefully cut rubber. (2) The water piston ought to be packed with tallowed hemp for cold water, bronze for hot water. (3) The packing of the stuffing boxes with glands, piston rods, ought to be made with a layer of fine asbestos. Such stuffing boxes ought to be screwed up as lightly as possible, and just enough to prevent escape of steam.

Inspecting the Plunger.—If the pump does not work properly, that

[1] To obviate intermittent feeding Koerting's automatic feeder is now used. Its constantly normal working is a real safeguard.—J. N.

is generally due to the plunger. It must be inspected, but it is not necessary to remove the plunger box; it is necessary to clean it as well as its seat, but it must never be filed. The plunger ought to be replaced in the direction in which it was taken out.

Lubricator.—The lubricator ought always to be open, and the jet which regulates the supply of oil ought always to be adjusted to the same mark so that there flows about two drops per minute. Lubrication must have careful attention, because on it depends the preservation and good working of the pump.

Feed Valves on the Supply Tank.—There ought, at least, to be one valve open on the feed even when the pumps are stopped. When it is desired to change the boilers, first the valve of the new boiler is opened and the old one closed, or both operations may be done simultaneously. From time to time it is necessary to make sure that the valves fixed on the return stroke of the pumps on the cast-iron feed, and those placed on the reheaters, work properly. One should feel each shock which they produce, each pulsation of the steam pump. If it were otherwise in the case of either it would be a proof that it was stuck and required inspection and cleaning.

Heated Surface of Steam Boilers for Use in Extract Factories.—The many years' experience of the authors, acquired in different tannin factories, enables them to affirm that the heating surface of the steam boilers is always represented by 5 square metres (54 square feet) in factories working by double effect and 4 square metres (43 square feet) in factories working by double effect—in both cases per metric ton of wood treated.

Different Furnaces and Gazogenes (Gas Generators). Bonnet-Spazin's Gazogene Furnace.—This system, shown in Fig. 67, is really a Faye's furnace improved by the addition of an actual combustion chamber, of a front armature to regulate the heat B behind the furnace A, and a manhole, G. As used to burn intractable fuel, such as exhausted wood chips or shavings from tannin factories, lignite, turf, etc., it must not be forgotten that the furnace in question is a sort of gas generator (gazogene) with its combustion chamber, its grate and its systematic feed-hopper, E, through which it is fed by the intractable fuel to be burnt, which clearly shows that the system as a whole depends upon a distillation of the intractable fuel, so as to produce inflammable gases, which burn in this combustion chamber with an intensity which develops an amount of heat which few furnaces of this kind produce, for it may be taken that 1 kilogramme of exhausted chips, with 60 to 62 per cent. of moisture, yields 1·4 kilogrammes of steam. This furnace, therefore, utilises wood so much the better as it distils it the more perfectly. Its

working is rational, by excluding any addition of coal to the fuel, because that paralyses the distillation by inducing the burning of the chips on the grate, and in the bulk of the woody mass in such a way that a portion of the combustible gases are already burnt up prior to their entrance into the combustion chamber, which diminishes the intensity of the heating of the boilers, under which this system of furnace is applied, by shortening the length of the flame, which ought to sweep about half round the boilers. Besides, the use of coal induces the formation of clinkers which attach themselves to the grate, which necessitates too frequent raking by the stoker, entrance of air and all the other evils inherent to the use of any other fuel than spent chips in this special case.

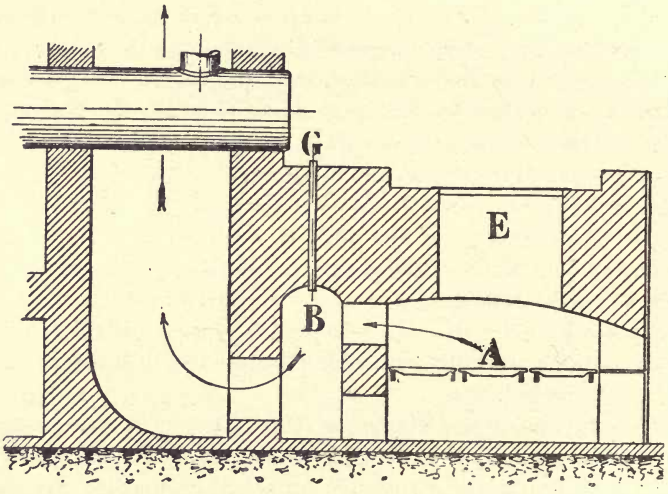

FIG. 67.—Bonnet-Spazin's gazogene furnace.

Practically, this system of gas generator (gazogene) furnace requires for normal working 3·5 square decimetres of grate per square metre of the heated surface of the boiler, 3·5 square metres per 100 square metres of the heated surface. Moreover, it is necessary that in all the parts of the boilers the section of the flues should not be less than $\frac{1}{7}$ to $\frac{1}{8}$ of the surface of the grate.

Working of Gazogene (Gas Generator) Furnaces.—If the masonry of the furnace be new it must be dried by keeping up a gentle wood fire for several days. Light the fire, working thus: Nearly close the main damper of the boiler to prevent too much draught. Spread on the grate a layer of readily inflammable fuel, shavings, paper, etc. Above this place a layer of well-dried twigs, and above this again some chumps of

dry wood. (In extract factories a stock of dry exhausted chips for use in lighting up and stimulating the combustion of the furnaces is always kept on hand.) The chumps of wood may either be thrown in through the furnace mouth or through the hopper. When the grate is charged the hopper is closed by an iron plate. Lay the fire on the lower bed in several places, simultaneously close the doors of the ashpit and open the furnace doors a little. Close the furnace doors when the whole surface of the grate is kindled and open those of the ashpit slightly. From time to time throw in chumps of wood—always dry—as the previous ones are burnt, and so keep up the fire. This heating with wood is continued as energetically as possible until the arch of the furnace and the combustion chamber are bright red. If need be the damper may be raised, but generally very little, to stimulate the combustion. When the combustion chamber is heated to quite its normal temperature one of the hoppers on the side is uncovered and charged with chips, care being taken to use rather dry chips. The fire is fed with chips through the first hopper and by chumps through the others for about half an hour, and when the first hopper is alight the same process is gone through with the other side hopper, then afterwards with the middle hopper if there be one on the furnace. When all the hoppers are filled with chips the main damper ought to be raised a little. The furnace is thus lighted up. The success of the operation depends especially on the heating of the masonry, which afterwards produces the distillation and inflammation of the chips. There need therefore be no fear in heating the furnace strongly before feeding it with chips.

Rules for Working the Furnace.—The chips should always cover the hoppers, to avoid access of air through them. The chips should descend continuously and regularly. Care must be taken to ascertain from time to time that no arches or vacant spaces are formed to prevent their descent and that only wooden bars are used as pokers. The ashpit doors ought to be always more or less open, the furnace doors closed, the valves of the latter generally open, the inspection pipes generally closed. The speed of combustion is regulated by opening the damper, but in a general way it ought to be always slightly open. These rules are only approximative ; they may vary with the draught of the chimney or the percentage of moisture in the wood. The inspection pipes ending in the combustion chamber indicate the intensity of the fire, and admit the complement of oxygen required for the inflammation of the gas in this part of the furnace. When the grate is in good working order, which is especially the case on starting work, the damper should be opened very little, the flap valves of the furnace doors closed and the

ashpits only half opened, for if the combustion be too much stimulated on the grate distillation is too rapid and the gases do not become inflamed. When the grate becomes encrusted, the damper must be raised a little, the ashpit doors opened gradually to a greater extent, the flap valves of the furnace doors opened, and, if need be, one of the furnace doors is left ajar to admit air above the grate. This latter precaution is especially necessary when the grate is greatly encrusted. When the furnace is working well no smoke should issue from the furnace, and in the gas chamber the fire should be bright red, without flame or smoke. When it is necessary to stimulate the fire to get up pressure the poker or shovel is thrust underneath the fuel to remove the scoriæ which obstruct the entrance of air, and the main damper is raised a little.

Night Fire.—To keep the fire banked up during the night the hoppers must be well rammed with a wooden poker so as to press down the fuel as far as possible. It is then necessary to tramp with the feet on these hoppers and to make a heap above each, which is also trodden down under foot so as to pack the chips tightly. The damper is almost completely closed, but so that the smoke does not issue above the furnace. The furnace and ashpit doors are completely closed. The night-watchman ought to ascertain every time that he inspects the boilers that the furnace fire has not entered into combustion and that the pressure of the boilers has not increased. In the morning, to stimulate the fire, the main damper is raised to its normal height, the ashpits opened as customary, and the poker or shovel thrust under the grate to let the ashes fall which have formed during the night and block up the grate.

Cleaning Out the Grates.—The grate takes about eight or ten days to get dirty, so that complete cleaning need only be done after that interval has elapsed. This cleaning is done in sections, by cleaning out one hopper every four or six days, for example. The cleaning is done as follows: The wood filling the hopper to be cleansed is tramped down so as to form an arch and stop the descent of the fuel; the fuel on the grate is allowed to burn out, which takes about ten minutes, and only the corresponding part of the grate is cleaned. During this time the descent of the wood down the other hoppers is forced a little, and as soon as the cleaning of the hopper is finished, the cleansed portion of the grate is covered with inflamed fuel from the neighbouring hopper; then when the fire has resumed its normal working the wood is made to descend as formerly through all the hoppers, poking through the arch formed in the beginning. It is necessary to select for cleaning a time when the pressure is high and when the consumption of steam in the different departments of the factory is low.

Cleaning of the Ashpit and the Heating Chamber.—Every morning all

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the ashes in the ashpit must be removed. After clearing it out the small tub in the ashpit must be filled to the brim with water. This tub ought to be filled with water two or three times a day and night, so that the ashes may be always drenched as soon as they fall from the grate. After each cleaning of the grate or part thereof the clinkers must be removed outside, and not piled in a heap in the heating chamber. This heating chamber ought always to be in good working order, and the gangway in front of the façades well cleared, so that the ashpit doors may be easily handled.

Godillot's Furnace.—For the combustion of intractable fuel, spent tan or exhausted chips, the systematic combustion furnace of Godillot seems to the authors to be perfectly adapted. A summary description will, therefore, be given of this system, which has received numerous applications, both in tanneries and in the tannin industry. To realise perfect and hence economical combustion in a factory furnace, the amount of air required to burn the fuel must be brought on to the grate and spread regularly over it. It will be seen, therefore, that if it is desired to use such fuel as moist tan chips, impregnated with water or residuals from extract factories, the largest of ordinary grates are defective, each charge blocking up the interstices through which the air passes, and almost extinguishing the portions in ignition. Take as an example of the poverty of these chips the residue from the manufacture of chestnut extract. The chips, as they issue from the decoction, contain 66 per cent. of moisture and 34 per cent. of woody fibre (cellulose), the calorific capacity of the woody fibre being half that of coal, 4,000 calories instead of 8,000, the 34 of woody fibre may be replaced by 17 of coal. This substance may, therefore, be compared to a fuel containing 17 of coal and 66 of moisture, or bringing the total to 100, 20 per cent. of coal and 80 per cent. of moisture. It will be seen that the ordinary grate is quite unfit to burn such fuel. Besides, intractable fuel is bulky. The following table gives an idea of the space occupied by each :—

TABLE XVII.—COMPARATIVE VALUE OF WASTE PRODUCTS USED AS FUEL

	A.	B.	C.	D.	E.	F.	G.	H.	I.	J.
Moisture . . .	68	52	62	40	55	60	14	12	29	65
Calorific intensity .	800	1,400	1,200	2,000	1,500	1,100	3,200	3,300	2,600	2,600
Weight (kg.) of cub. met. . . .	500	330	500	300	150	200	260	140	260	600
Kg. = 1 ton coal .	1,000	5,700	6,600	4,000	5,300	7,300	2,500	2,400	3,200	4,500
Cub. met. = 1 ton coal	20	17	13	14	35	36	10	17	12	10

A, wet spent tan ; B, drained spent tan ; C, chips from extract factories ; D, wet sawdust ; E, wet bagasse (cane sugar) ; F, wet cane chips from diffusion ; G, dry chips (oak) ; H, rice hulls ; I, linen waste ; J, peat.

The last horizontal line of the table shows the volumes in cubic metres equivalent to 1 ton of coal. Thus, to replace a ton of coal, it is necessary to burn 20 cubic metres of wet spent tan, 35 cubic metres of bagasse, or 13 cubic metres of exhausted chips. To accomplish this result Godillot designed a pavilion grate in the shape of half a cone. It consists of horizontal, semicircular bars, the diameters of which decrease from base to summit. These bars overlap like the laths of Venetian blinds, so as to retain the finest particles, whilst allowing the necessary space for air to pass through. Hand stoking of these enormous bulks would be too severe a task. The mechanical stoking

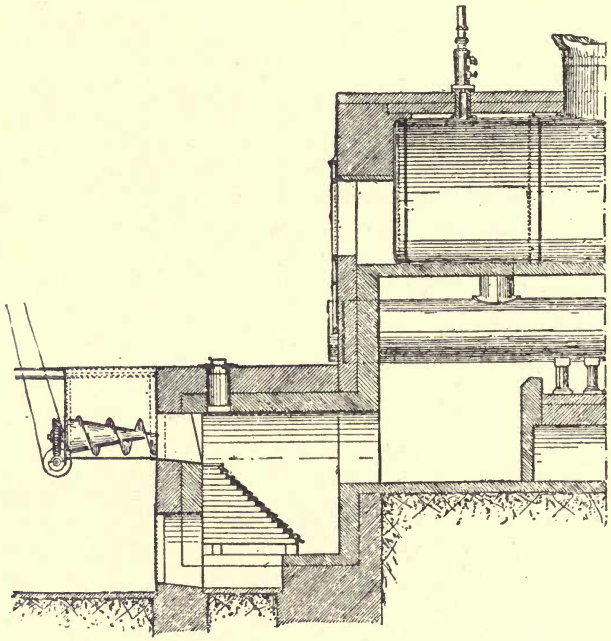

FIG. 68.—Godillot's furnace applied to a semi-tubular boiler.

is done by means of a cast-iron helix with increasing "buckets" (*augets*) [expanding screw]. Woody fibre is in the form of irregular, filamentous fragments; spent tan and bagasse do not run readily, because the particles descend for certain on to the threads of the helix and cannot get freed therefrom. The cone of the helix, instead of being cylindrical is conical, and the point is directed towards the exit, so that the space between the threads of the helix shows an ever-increasing capacity. In this way the helix can draw along the material throughout the whole length of the hopper, at the bottom of which it turns. Fig. 68 shows the plant used by Luc and Patin, manufacturers of

oak extract at Nancy, where chips are burnt with 62 per cent. of moisture. The substance to be burnt is run into the charging hopper; the helix, with increasing thread, brings it to the top of the pavilion grate; it dries, heats, inflames, descends on the slope of the cone in the form of a thin layer, in proportion as the fuel underneath is consumed; finally, it reaches the horizontal grate, where the combustion is finished, and where the ashes accumulate; they are easily withdrawn by the side doors for the purpose. The furnace works quite regularly. Luc and Patin's plant comprises seven furnaces, heating 500 square metres of heating surface; the economy realised is 18 tons per day.

One kilogramme of drained (*essorée*) spent tan, with 55 per cent. of moisture, barely converts 450 grammes of water into steam, in ordinary plant, whilst in experiments made by M. Compère, engineer-director of the Parisian Association of Steam-boiler Proprietors, on Godillot's plant, 1,700 grammes were obtained. Godillot has erected a similar installation for six boilers, 500 square metres of heating surface, at the Gondola factory at Nantes, likewise for the Company of Fives-Lille, for the factory of Di Vono-Pringo (Java), for the factory of Trystam & Co. at Dunkirk, with the same success. The advantages of the plant are the following: (1) The raising of poor material to the rank of industrial fuel; (2) better combustion; (3) regularity in working, which avoids the risk of overheating and allows of a regular draught; (4) elimination of access of air; (5) simplification in stoking; (6) complete prevention of smoke, even in the case of most smoky fuel.

Chimneys. Brick Chimneys.—There is an advantage in a tannery having a brick chimney lined at top and bottom; it should be furnished with a lightning conductor, and its interior fitted with access ladders, because this kind of industry does not require great regularity of pressure in the boilers, even if it utilises its spent tan as fuel, using a gazogene furnace, which requires a chimney section of 64 square decimetres or 900 millimetres of diameter at the top per 100 square metres of heating surface of the boilers.

Prat's Chimney with Forced Draught.[1]—Extract factories, of which the output and the regular working of the manufacture are a function of the pressure of the steam, have a greater interest in installing a chimney with forced or mechanical draught, which consists in propelling into a special apparatus, termed "the pressure transformer," a current of air under pressure, generated by a fan, placed outside the circuit of the gases to be entrained, and which generates by its passage an induced current which entrains the combustion gases. This

[1] Now advantageously replaced by Sturtevant's system.

transformer may either be placed in the interior of the chimney, to increase the draught, which may increase from 2 to 25 mm., or directly in the main flue, where the combustion gases join, where it then does the duty of the chimney. Fig. 69 shows in section the principle of this system, which has since 1896 received numerous applications, and

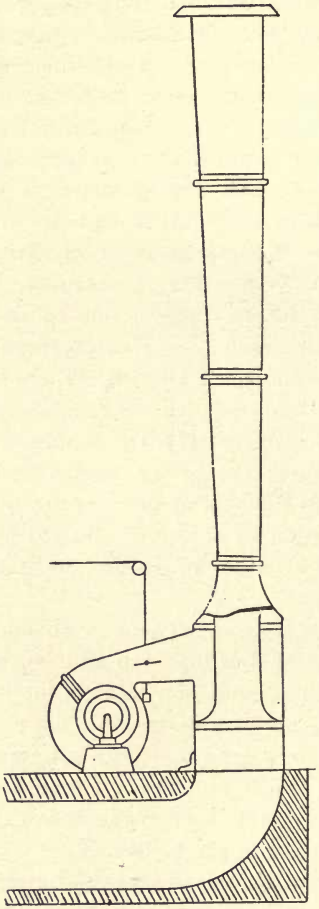

Fig. 69.—Mechanical draught applied to a furnace.

which consists in the aggregate of: (1) a centrifugal fan (blower) placed outside the circuit of the combustion gases, so as to avoid simultaneously its oxidation by sulphurous acid, or its encrustation by soot, and its heating by gases, generally at a high temperature; (2) a transformer of pressure forming a chimney; (3) a vent pipe, with regulating valve, connecting the ventilator with the transformer.

These appliances, in view of their maximum utilisation, are calculated and constructed on a practical basis by which it is possible to obtain (1) a better yield of the pulsating fluid; (2) the free propulsion of the gases aspirated. It may be said that the draught in constant working, which can be realised with a motor power of about 1 per cent. of that produced by the boilers, by means of a simple fan placed outside the circuit of the combustion gases, with the pressure transformer adjusted to the orifice, in which it is to be used, and with the autovariable depression which results therefrom, has not only great aspiration capacity, allowing of its utilisation in all degrees of intensity of combustion, whilst rendering heating easy and economical, but it still further presents a real superiority over fans which aspirate or directly propel the combustion gases. This system of draught by suction maintains its superiority, both as regards working expenses and general working, even over ordinary chimneys with natural draught. Thus it may be pointed out that an extract factory, treating 60 tons of wood per twenty-four hours, consequently requiring 240 square metres of boiler surface and 8·4 square metres of grate (in the case of a gazogene furnace), will constantly require a natural draught chimney of about 30 metres in height and 1·4 metres of diameter (1·5 metres in section), which would be replaced by a chimney with mechanical draught on Prat's system of 10 metres in height only. In the case of a Godillot furnace (pavilion grate) and for a boiler of 100 square metres of heating surface, it is necessary to take 62 square metres of section or 800 mm. in diameter as the section to be given to the chimney.

Steam Engine.—It will be sufficient to point out that the choice of a steam engine in an extract factory is not of great importance, the escaped steam being invariably and totally utilised in heating the evaporation plant and for the concentration of the tannic liquors. Nevertheless, it will be well preferably to adopt a horizontal system, with condensation and expansion capable of being varied by the regulator, revolving at the rate of 60 to 100 revolutions; this arrangement allows in the first place of sure and constant working, at the same time that the facility of economic working may be by escape, or, if need be, by condensation. Fig. 70 shows Robatel, Buffaud & Co.'s type, of which the regular working, the solid construction, the simple management, the easy installation, and the maintenance, which costs nothing, have caused it to be adopted in many factories.

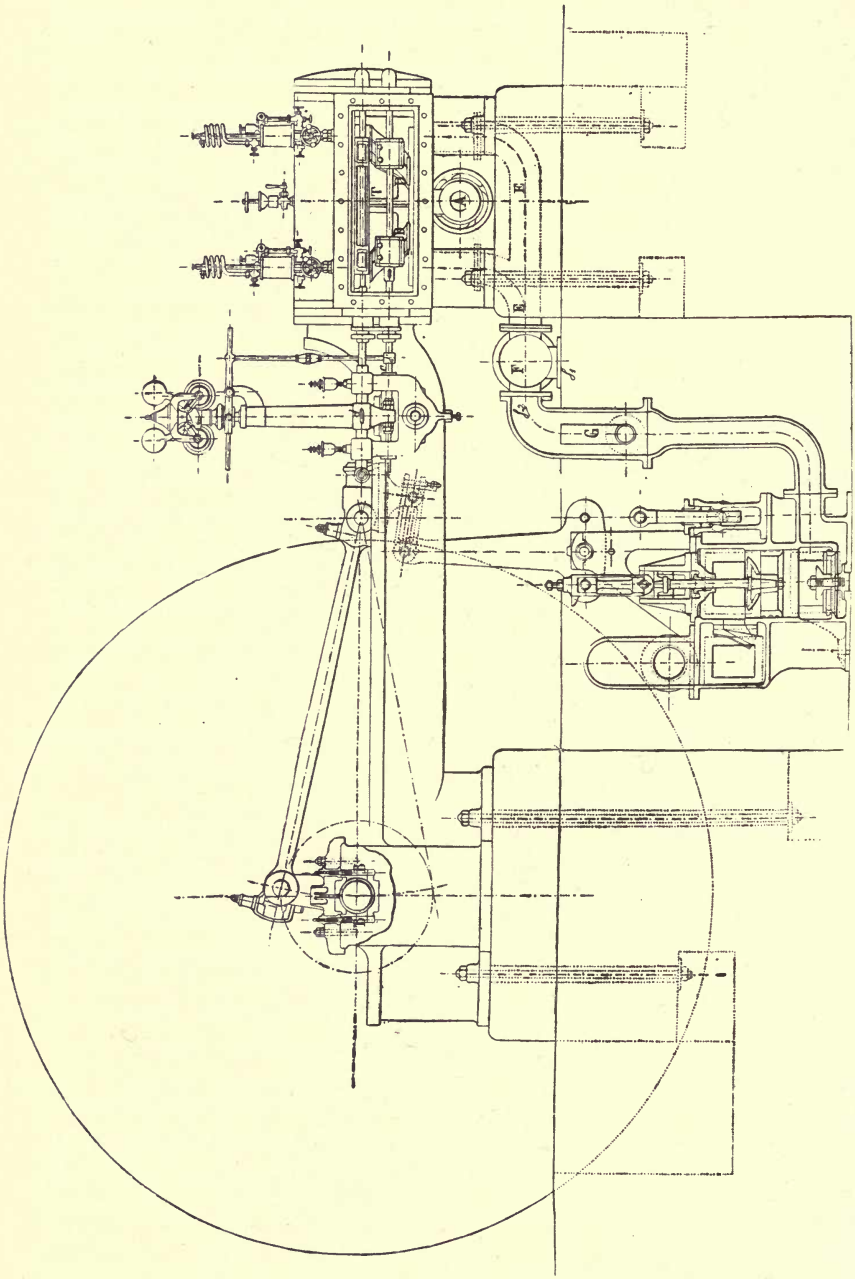

FIG. 70.—Condensation steam engine. Robatel, Buffaud & Co.

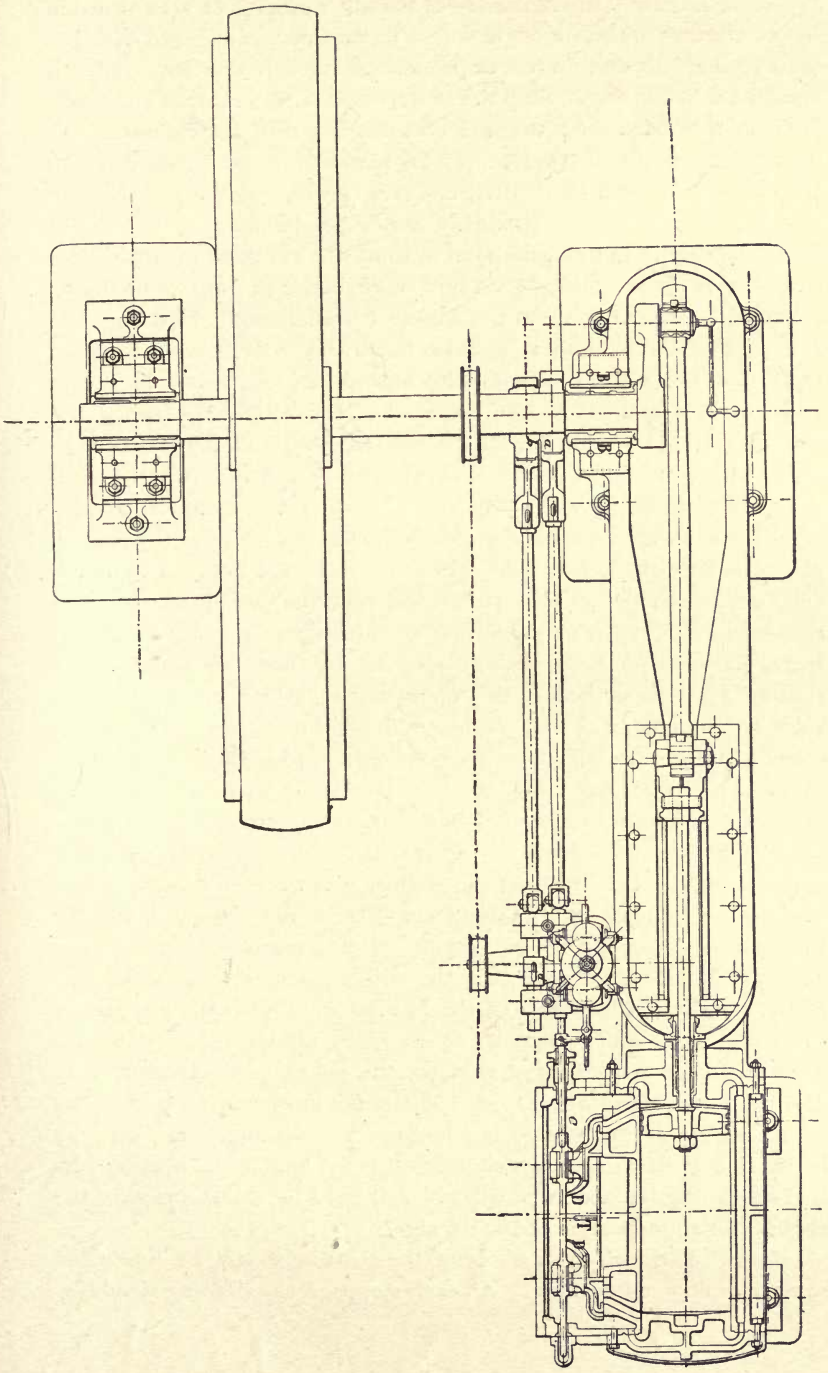

FIG. 70A.—Condensation steam engine. Robatel, Buffaud & Co.

Steam Engines. Maintenance.—Ascertain regularly on each occasion before starting the steam engines that no workman is engaged with the belts or shafting, and no tool or material of any sort that may cause an accident is in the way. Stop the steam engines preferably at the dead-point so as to have the piston rod completely out of the cylinder. To start an engine it is necessary: (1) To open widely the steam valve on the steam feed from the boilers, but very slowly, especially if the pipe is cold, so as to heat it gradually and avoid bumping of water; (2) bring the engine to the dead-point in front if it is not so already; open the blow-off cocks of the steam box and cylinder in front and behind; (3) open the steam valve on the engine to reheat and "blow off" the rear part of the cylinder; (4) close the steam valve, turn the engine by hand to bring it to the dead-point behind, and again open the steam valve to heat and "blow off" the front of the cylinder. During the time of heating the cylinder, fill the lubricator with oil, lubricate all the joints which only possess one lubricating hole and regulate the automatic lubricators with visible flow; (5) when the cylinder is completely warm and the lubrication finished, close the valve of the engine, place the latter by hand at its starting-point, that is to say, a little beyond one or other of its dead-points, and open the valve gradually until it is completely open in proportion as the engine assumes its speed. Leave the blow-off cocks entirely open for the first two minutes after starting, then close them. To stop an engine slowly, close the steam valve on the engine as well as the automatic lubricators. The engine ought to be stopped at one of its dead-points, preferably at the front one, and if it does not come into that position it must be brought so by hand. Ascertain before starting the steam engines that the butterfly valve of the escape is open to the free air if the evaporating plant is not at work. For lubricating the engines it is necessary to use valvoline for the cylinder and the draw box (a thick and black oil), and for the bearings, crank, eccentric, regulator, etc., machinery oil of good quality. Make sure always that the automatic lubricators work perfectly, and keep the glasses of the lubricators of the cylinders and of the draw boxes quite clean so as to verify always the flow of oil. Engines ought to be kept very clean, the polish parts cleaned every day, the rough parts well wiped. When the mechanic finds that one of the organs of the engine has become hot, he ought to force the lubrication of that organ, watch it attentively, and if the heating persists advise the foreman, who will order, if need be, the stoppage of the engine and an inspection of the damage.

General Shafting.—The whole of the shafting should be lubricated every morning and evening. At each stoppage the shafting should be

inspected to see that no bearing has become heated; as soon as it is perceived that one of the bearings has a tendency to become heated, it must be closely watched and its lubrication forced up to the next stoppage. If need be, stop at once and lift the cap to see that there is no obstruction on the bearing or the shaft; should there be any trace of obstruction, remove it immediately before starting again, and take care to clean the lubricators and then the lubricating holes and the little grease channels in the shaft cushions.

Fixed Pulleys.—In the case of all pulleys in two pieces make sure that all the pins are perfectly screwed to the block and that the collars have no play. If it be seen that any of the pins are unscrewed stop the shafting at once and rescrew these pins; rescrew the collars immediately if need be.

Loose Pulleys.—It is necessary when inspecting the bearings to inspect the loose pulleys at the same time and insert lubricant in their lubricator and see that they do not heat. If one begins to heat, the shafting must be stopped, the lubrication of that pulley forced and restarted. If the heating persists, which is a sign of obstruction, it is necessary to undo the shafting which carries this pulley; remove the obstruction, clean the lubricator, the lubricating holes, and the little grease channels of the locking ring.

Piping and Taps in General.—Each department where steam is used having its special valve for drawing steam from the boilers, this valve should be perfectly open when the corresponding department is at work and completely closed when it is stopped. When the valve on the boilers is opened it must be done very slowly; first of all only run on a thread of steam to heat the pipes throughout its length, and gradually open afterwards to avoid all bumping, which would cause the joints to bump and the pipes to burst. All the valves and all the taps ought to be kept constantly in perfect working order, the stuffing boxes and the joints quite tight; all leakage ought to be noted and repaired forthwith, however small it may be, as soon as it occurs. The joints of the pipes and the steam taps should be made with best quality of sheet-rubber of 4 to 5 mm. thick with brass-wire gauze insertion. The packing of the glands of the valves and taps ought to be made with a layer of asbestos of the desired size, and these glands ought to be packed as little as possible, only enough to prevent any escape of steam. Take care in all the taps to screw the pins of the glands very equally so as not to wedge them.

Elevators and Conveyers. Ewart's Chain Elevator.—This system consists (Figs. 71 and 72) of links which may be aggregated together and

detached at will. The links are generally rectangular, three of the sides are cylindrical, and the fourth consists of a hook. This chain is of malleable cast-iron, of quite a special quality; all the links are rigorously calibrated, and consequently interchangeable, which assures regular working, along with the greatest facility for elongating and shortening the chain at will without loss of time. Although Ewart's chain is made of metal, it is not, for equal effort, heavier than a belt; in many cases it may even be less.

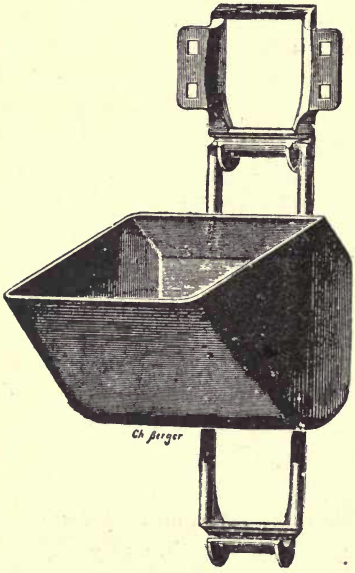

FIG. 71.—"Ewart's" chain.

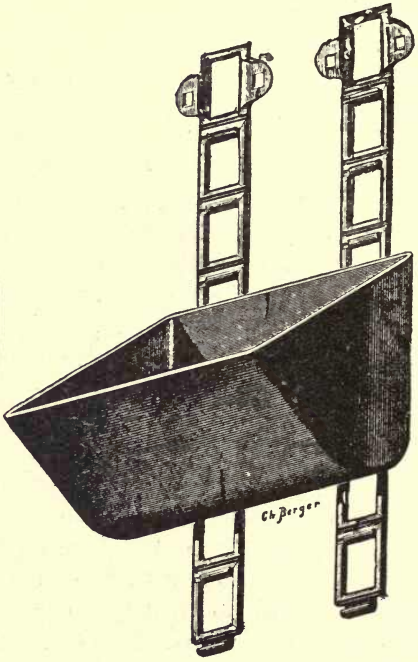

FIG. 72.—"Ewart's" chain coupled.

Harrison's Chain System.—Harrison's chain consists of closed links and open junction links. Fig. 73 sufficiently shows the method of connecting up the chain. Like Ewart's chain, Harrison's steel chain is applied to elevators, conveyers, etc., but it is more particularly adapted for plant that has to do heavy work, or where the conditions of resistance of cast-iron would involve the use of two coupled Ewart chains, or at least a strengthened and consequently a heavier chain. Harrison's chain was first made of cast-iron, but the simple and regular form of the links led to its manufacture in cast-steel, which has placed it in the first rank of chains with detachable links. At the present day the firm of Burton Fils, of Paris, who are specialists in this branch of con-

struction, manufacture this chain in stamped steel, which still further increases its durability and its resistance to rupture.

Plummer Blocks. Cast-iron Feed Receivers.—Figs. 74 and 75 show these

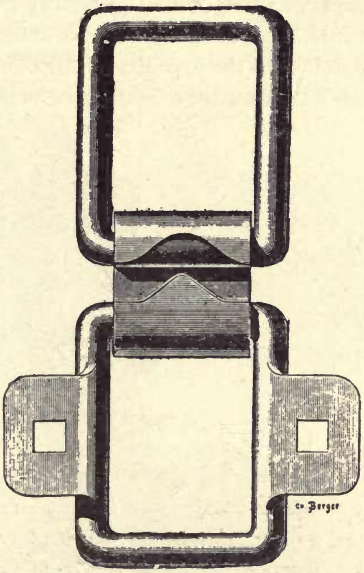

FIG. 73.—"Harrison's" chain.

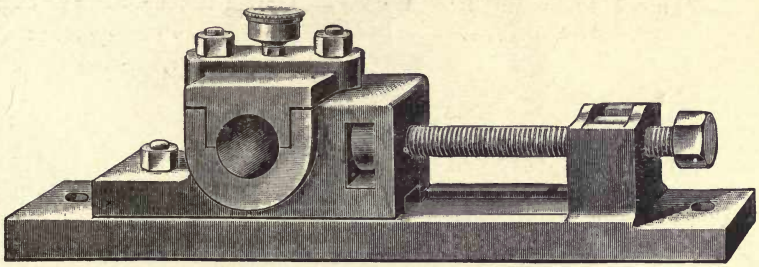

FIG. 74.—Plummer block.

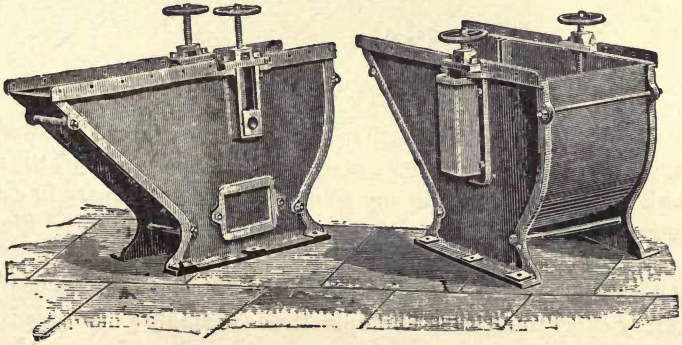

FIG. 75.—Cast-iron receiver.

accessories which complete with the chains and the stamped sheet steel cups the whole elevator shown in Fig. 76.

Conveyers.—These appliances, which generally receive the chips of wood or ground bark from the elevators, consist of an endless cotton band, of dimensions corresponding to the capacity of the elevators, of two rolls, or steel drums, the one a transmitter of speed, the other a receiver, and of

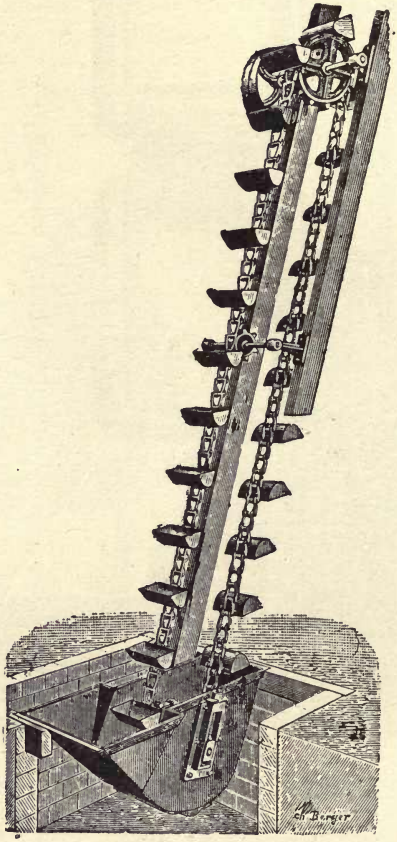

Fig. 76.—Elevator.

some sloped rolls along the length so as to guide the edges of the band. A conveyer may also be fitted with a movable truck, which enables the chips or bark to be discharged, in any point of the wood granary in extract factories, or in the warehouse for crushed bark in tanneries. Figs. 77 and 78 show these two applications.

Chip Granary.—The men allocated for filling autoclaves ought to use every effort to conduct the operation as rapidly as possible; they

ought to begin to shoot the chips as soon as the signal is given by the battery foreman. During the time which elapses between the two charges they ought to accumulate as much wood as possible on the aperture of the autoclave. The chips must not remain more than three or four days in the granary, as they are liable to ferment; it is necessary therefore to empty this granary every four days, at least, by collecting together all the chips from the far corners. The men ought to remove

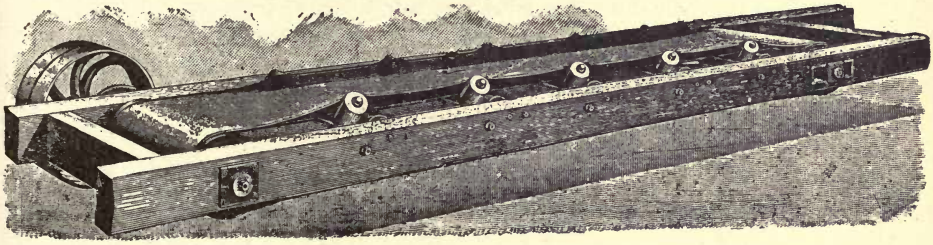

FIG. 77.—Conveyer.

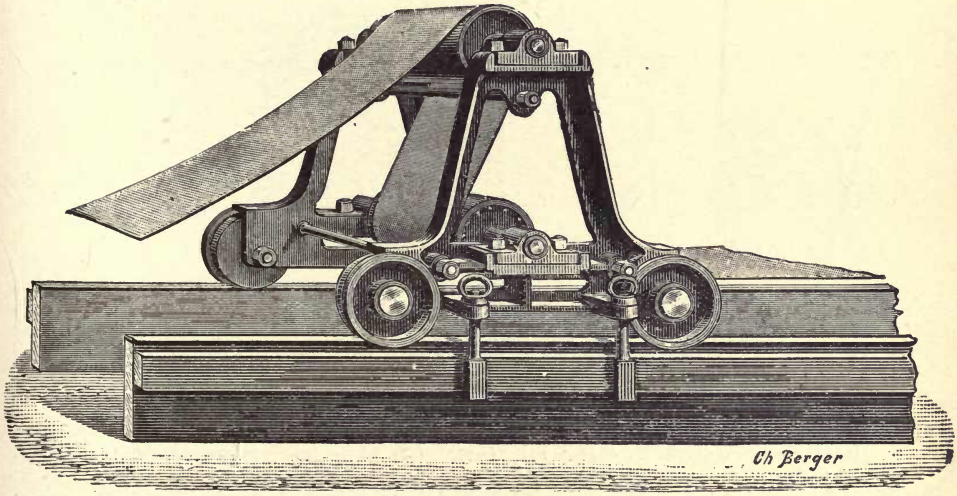

FIG. 78.—Portable truck of conveyer.

carefully all pieces of iron, large or small, which may be mixed with the chips. No piece of iron, however small, should ever enter into the autoclaves.

Cutters.—Only such industrial machines will be mentioned as are of great capacity, and consequently turning between 350 and 450 revolutions a minute, slow-speed cutters (100 turns a minute) with a long passage and mechanical pusher being now almost completely abandoned, as absorbing much of the motive power, in consequence of the pressure

of the log against the sides of the boss (*tourteau*), thus playing the part of a real brake, without any compensation in the output. It may be added that for the same power absorbed a cutter with an angular boss of great speed, such as that shown in Fig. 79, has an output twice as great as that of the cutter with mechanical pusher. The high-speed cutter of great capacity is moreover very simple (see Figs. 79 and 79A). Its organs are reduced to: (1) One angular boss of cast-iron 700 to 800 mm. in diameter fitted with two apertures or bays for receiving the steel knives or blades, the number of which varies according to the capacity of the cutter, and which are fixed by means of pins or gudgeons

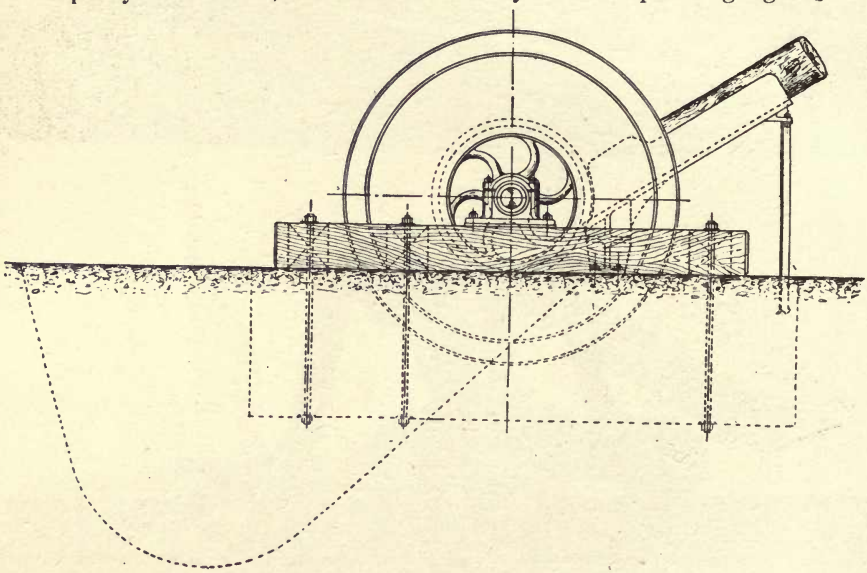

FIG. 79.—Cutter of great capacity.

reinforced by screws; that is the part of the construction that ought to be strong enough to stand any test. (2) One steel shaft traversing the centre of the said boss on which it is strongly fixed. (3) Three lubricating bearings with bronze cushions. (4) One inclined cast-iron passage for reception of the logs, and its spur or anvil which assumes the shape of the angle of the central boss (*tourteau*), which facilitates the cutting. (5) Two pulleys loose and fixed.[1] (6) One cast-iron flywheel, the whole protected by a hood of steel plate and mounted on three oak beams, tied and bolted, which rest on a concrete foundation, at the bottom of which is a pit for receiving the chips and into which dips the cup elevator previously described. A cutter putting through

[1] Unless Burton's gearing be used.

2,250 kilogrammes (say 2¼ tons) per hour of logs 25 cm. (say 10 inches) square absorbs 20 to 25 h.-p. In normal working the log once placed in the sufficiently inclined shute ought to be drawn mechanically by the blades of the boss. The workman in charge of the cutter ought to have nothing else to do but to feed in the log as soon as the former one is "swallowed". Chopping machines and bark crushers need only be mentioned in passing, the systems being numerous and described in several works on tanning[1] and the illustrated catalogues of French and German constructors of tannery machinery, such as Berendorf, Allard, Moenus, Ste. de Durlach, etc.

Instructions Regarding Cutters and Elevators. Starting.—Before starting a cutter see that there is oil in the three bearings. Begin by

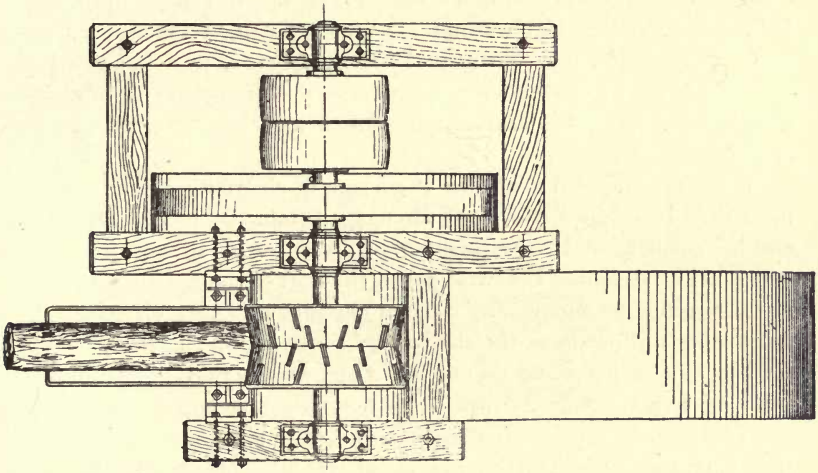

FIG. 79A.—Cutter of great capacity.

starting the elevator. This operation requires a man to put the belt on the elevator and another at the cup chain ; the latter ought to pull on the chain, especially if there is wood in the bottom pit, which is liable to block the cups and burn the belt. The cutter is then started, by slowly pushing the belt of the loose pulley on to the fixed pulley.[2] The belt must be brought into gear gradually. Before placing the logs of wood in the cutter, it is necessary to see that the boss has attained the right speed, and that the elevator has completely emptied the pit of chips. When such is the case, the cutter may be fed with logs. As far as possible, large logs should be alternated with small ones. If, owing to the large size of a log, the speed of the cutter

[1] *La Tannerie*, L. Meunier and C. Vaney, 1903.

[2] It is better to use friction gearing.

slackens, it is necessary to wait until it has regained its speed before running in the next log. During working, if the elevator stops it is necessary to cease forthwith feeding it with logs, and not to restart that elevator until after the cause of its stoppage has been ascertained. If need be, inform the foreman.

Stopping.—To stop, first cease feeding with wood, take the belt out of gear, leaving the elevator at work until it has emptied the pit of chips. In the interval clean up around the cutter, and remove the chips projected therefrom or which have fallen from the elevators. Then bring the elevators into gear. No chips must be thrown into the pit when the elevator is not at work.

Lubrication and Heating.—Oil must be run into the lubricators of the bearings at least every two hours, and it must be ascertained frequently during working that the bearings have no tendency to abnormal heating. If one of the three bearings, particularly the central one, becomes somewhat hot with a tendency to increase, it is necessary to lubricate freely at first; stop the cutter a moment to allow the bearings to cool, and restart.

If the heating still continues it is a sign of obstruction. The cutter must then be stopped, the shaft disengaged from the heated bearings, and the cushions of the bearings scraped with the scraper on the parts which are scratched. Likewise polish the part of the shaft which rests in the bearing, to remove any trace of obstruction. Carefully clean the lubricators and put back the shaft in its place. Run the cutter empty for, say, half an hour, and do not insert any wood until it is seen that no fresh heating occurs. At each stoppage of the shafting of the cutters it is necessary to give a screwing-up turn to the lubricators of the loose pulley, and fill these lubricators with special lubricant every day.

Sharpening the Knives.—The knives should be sharpened very uniformly, so that the cutting edge is perfectly straight and set square on the side.

Inserting and Adjusting the Knives.—The knives ought all to be inserted in the same way and they all ought to project 4 to 5 mm. from the central boss (*tourteau*). It is necessary to use the special gauge prepared for the purpose and to follow it very exactly. Take care to screw up very tightly the bolts which fix the knives.

Regulating the Spur of the Chute.—The spur ought to be securely bolted to the chute, and the position of the latter may be varied by screw bolts fitted to the cross beam of the building. This position must be so adjusted that the play between the spur and the central boss (*tourteau*) is 7 to 8 mm. quite uniformly throughout all its length. When the feather edge of the spur is rounded by the friction of the

wood, it must be redressed to bring the feather edge to a sharp angle. Such work is done with a graving tool. When, by repeated dressing, the spur is shortened to such an extent so as to leave only 7 mm. (0·275 in.) of play between the central boss (*tourteau*) and the spur, the latter must be changed.

Extraction. Diffusion Plant. Wooden Vats.—The tannin industry, which in the beginning used wooden vats as extractors, could profitably use them still for the three reasons previously given: A good yield, easy clarification and cheap decolorisation, high strength in tannin. As to durability, pitch-pine vats with staves 78 mm. (say 3 inches) thick will last as long as copper autoclaves. The authors have handled vats of this kind which, after fifteen years' use, taken down and displaced three times, continued to be used for the safe extraction of sumac and mimosa without their tightness being afterwards compromised. Did not the Gondolo Factory, a firm of reputation, start with wooden vats? a certain arrangement of which vats was the subject of their patent (*brevet*) of 2nd June, 1880. Besides, this important factory, which treated as much as 300 tons in twenty-four hours, possessed numerous batteries of wooden vats to do the work of which the catalogue of plant and equipment of 1903 again makes mention. At the present day, five French factories and some foreign ones still use, with success, this rational form of extractors. The authors recommend this system, so much the more because, installed according to their instructions, it presents all the advantages of a battery of copper autoclaves. The factories which make extracts other than chestnut have every advantage in the exclusive use of wooden vats (except their intrinsic value) without any of their disadvantages, and for the following reasons: (1) With a battery of wooden vats no more steam, if not less, is used than with a battery of autoclaves. (2) The battery of wooden vats has the advantage over copper vessels of being absolutely safe. (3) The installation of a battery of wooden vats costs less as initial expenditure and requires less repairs than a battery of autoclaves, because the pressure of the liquor demands special and costly piping which soon leaks. (4) The management of a battery of wooden vats, although more onerous than a battery of autoclaves, is more simple and more systematical. (5) The yield per cent. on the wood treated in extract of 25°, obtained by the use of wooden vats, is equal if not superior to that furnished by a battery of autoclaves. (6) In open vats heated to 100° C. the juice or liquor obtained is less coloured and contains less insolubles (consisting of particles of wood mechanically entrained, pectic and resinoid bodies), consequently more quickly clarified and more readily decolorised afterwards than in the case of juice or liquors extracted under the obligatory

pressure of the autoclaves. (7) Finally, and it is the most important point, after having demonstrated the superiority of extraction in open wooden vats, this system yields extracts with a high percentage of tannin, which always exceeds by 2 per cent. that of extracts obtained by extracting the wood in autoclaves, the temperature, a function of the pressure, which in such plant reaches 1·5 kilogrammes (equal to 127° C.), destroys by that fact alone a notable quantity of tannin. In this connection it will suffice to place, in parallel, the table summarising the experiments of Professor Eitner, an authority on the subject, whose results fully con-

TABLE XVIII.—EITNER'S EXPERIMENTS ON THE EXTRACTION OF TANNING SUBSTANCES AT DIFFERENT PRESSURES

	A. Extract per cent.				B. Tannin.				C. Non-Tannin.			
	Pressure.[1]				Pressure.[1]				Pressure.[1]			
	1.	2.	4.	6.	1.	2.	4.	6.	1.	2.	4.	6.
Barks: 1. Pine	29·06	32·76	31·54	30·72	16·24	12·92	8·58	6·49	12·82	19·82	22·96	24·23
„ 2. Oak	22·14	23·79	23·88	24·04	11·07	7·99	5·62	3·22	13·07	15·80	13·26	20·82
„ 3. Cajota	40·54	41·41	33·14	26·38	21·45	21·45	11·41	2·27	18·79	20·00	21·73	24·11
„ 4. Mimosa	42·10	45·00	43·52	41·33	31·61	30·75	29·98	26·60	10·49	13·54	14·25	14·73
„ 5. Hemlock	13·70	14·07	13·73	12·70	9·30	8·34	4·50	2·13	4·40	5·73	8·20	11·60
„ 6. Willow	9·16	14·95	17·49	19·39	4·80	3·16	1·59	1·59	6·0	10·15	15·90	17·80
Myrobolams	41·73	44·19	45·88	44·12	25·02	23·02	14·52	12·49	16·12	21·17	31·46	31·63
Algarobillas	68·62	63·06	49·25	48·85	36·44	24·04	8·37	8·47	32·18	39·04	40·88	40·38
Divi-divi	69·40	64·72	55·90	46·56	45·12	33·14	18·08	14·93	24·28	31·58	37·26	31·63
Valonias	49·23	50·70	47·79	41·45	29·97	27·28	24·78	18·92	19·26	23·42	23·41	22·53
Galls	45·24	43·87	41·43	39·90	29·32	27·08	23·78	17·73	15·92	16·78	17·65	18·25
Sumac	43·38	52·33	51·10	47·48	22·85	22·70	11·27	8·87	20·53	29·63	39·83	39·61
Oakwood	9·76	10·96	23·60	24·81	6·44	6·50	5·52	2·57	3·32	4·46	18·08	22·34
Quebracho	23·91	24·38	25·39	26·23	21·05	21·50	18·42	13·60	2·86	2·88	6·96	12·63
Tannin	100·12	100·59	100·16	92·79	94·76	85·55	63·49	39·14	5·46	15·04	36·67	53·05

firm our assertions. The substances experimented on were placed in an autoclave and submitted for two hours to temperatures of 120° C. (248° F.) = 1 atmosphere; 133° C. (271·4° F.) = 2 atmospheres; 151° C. (303·8° F.) = 4 atmospheres; 164° C. (327·2° F.) = 6 atmospheres. The results of these experiments are given in Table XVIII., which shows in A the amount of dry extract per 100 parts of tannin examined; in B the proportion of tannin contained in this dry extract, and finally in C the proportion of non-tannin in the same residue. It follows from these experiments (1) that the strength of the extract, which increases from 0 to 2 atmospheres with about two exceptions, decreases afterwards in the greater number of cases or maintains its strength between 2 and 4, to

[1] In atmospheres.

fall very greatly between 4 and 6 atmospheres. (2) In all cases the tannin per cent. of substance treated lowers rapidly in passing from a pressure of 1 to 6 kilogrammes. (3) Between 1 and 2 kilogrammes, and consequently between 120° and 133° C. (248° to 271·4° F.), the decrease in tannin is generally quite small, especially with cajota and mimosa bark, oak and quebracho woods, but with algarobilla, divi-divi and myrobolam the loss in tannin is considerable. (4) The non-tannins of Column C always increase with the pressure, owing, on the one hand, to the transformation of tannin into non-tannin, and, on the other hand, to the solution of these bodies owing to their hydration.

Entrusted lately with starting a chestnut extract factory in Le Gard, in bringing the daily production from 2,500 kilogrammes (2½ metric tons) to 6,500 kilogrammes (6½ metric tons) of extract of 25°, the authors determined the difference in tannic strength of two extracts which were then manufactured. The one, from a battery of six open wooden vats, showed an average of 30 to 31 per cent. of tannin, determined during a course of manufacture of more than a month, and an average daily production of 2,500 kilogrammes (2½ metric tons) at 25°; whilst the battery of autoclaves, in which the pressure oscillated between 0·5 and 1 kilogramme, the daily production of which reached 4,000 kilogrammes (4 metric tons) of 25° extract, never exceeded an average of 28·5 per cent. of tannin. In fact, according to numerous analyses made on the two qualities of extracts, a minimum difference of 2 per cent. has always been to the credit of that made open to the air. These industrial experiments, based on the above data, enable the authors to maintain their preceding conclusions without fear of being crazy.

Battery of Wooden Vats.—Fig. 80 shows the authors' arrangement for a battery of sixteen wooden vats of 12,000 litres (2,640 gallons) capacity and capable of containing 3 metric tons of wood, answering, therefore, all the requirements of rational extraction.[1] In this system the heating surface required for the boilers will always be taken as 4 square metres per ton of wood treated in a factory possessing a triple effect; that is to say, that possessing such an installation, not 1 kilogramme of coal will be burnt, but there will be an excess of chips sufficient to light the furnaces after any stoppage, say weekly, for example. This battery is furnished with its piping and connections, which enable it to work rationally with the facility of isolating each vat. The transference of the juice or liquors is effected either by the weight of these pumped into an elevated vat, *i.e.*, by gravity, or by

[1] Especially suitable for tropical countries where plant is dear and tanning substances are extracted which gain by treatment in open air.

means of a bronze centrifugal pump[1] connected to the vats by two collectors, one for discharging, common to the transfers, and the other for filling, common to the water feed. Each vat is also fitted with appliances for steam heating, with two bronze discharging cocks; the top part is covered by a lid with a chimney for the evacuation of mud, and finally with a special water-level. The lower part of each vat has a double bottom and an arrangement for constant circulation, so as to accelerate maceration by the rapid washing of the slices from top to bottom.

Open Copper Pans.—The authors only prefer pitch pine for economical reasons, otherwise copper vessels may be substituted, but as there is no industrial advantage, the subject need not be dwelt upon further.

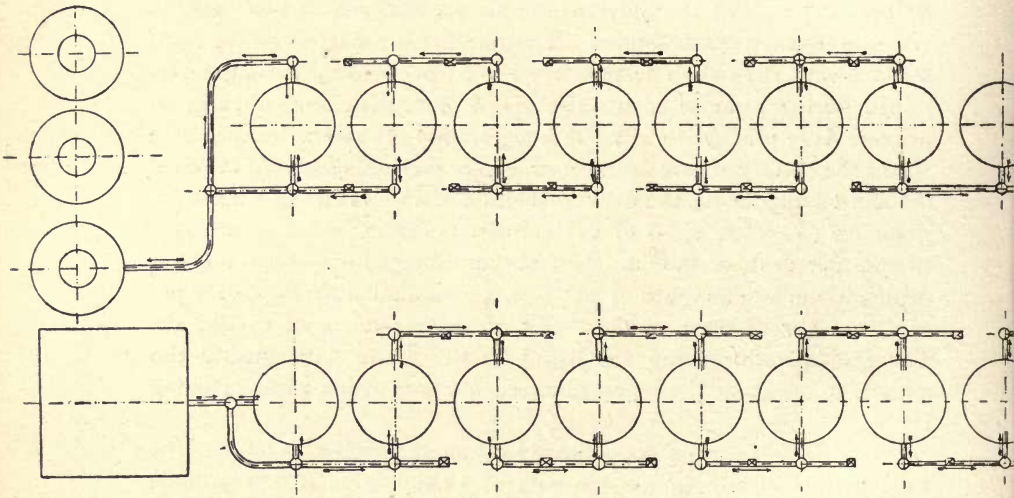

FIG. 80.—Battery of wooden vats for extracting tanning substances from chestnut wood (Noyer).

Copper Autoclaves or Extractors.—Practically, an extract factory can work with an extraction battery, consisting of four, five or six autoclaves, the useful capacity of which is generally from 10,000 to 11,000 litres (2,420 gallons), that is to say capable of holding $3\frac{1}{2}$ to 4 metric tons of wood chips, and yielding 5,500 to 6,000 litres (1,210 to 1,320 gallons) of liquor. It may be stated once for all that a tannin concern, to be of any importance at the present day, must treat at least 60 tons of wood per day.

Arrangement of the Battery.—Fig. 81 shows a battery of five autoclaves with their feed water, steam and juice pipes, as well as their

[1] Limb's dynamo pump.

taps; they are all adapted for the maceration of chestnut, and in the case of a battery of six autoclaves the sixth isolated is used for the direct extraction of quebracho or other mixed extract, which will be readverted to in Chapter VII. (Quebracho Extract). These autoclaves of the capacity above mentioned have the following dimensions: total height 4·6 metres, height of the cylindrical part $3\frac{1}{2}$ metres, exterior diameter 1·85 metres. The thickness of the copper plate of which they are made is generally from 8 to 9 mm. Per day of twenty-four hours fifteen to sixteen autoclaves are emptied; as it is necessary to deduct from the time required for boiling (*cuite*) that occupied in

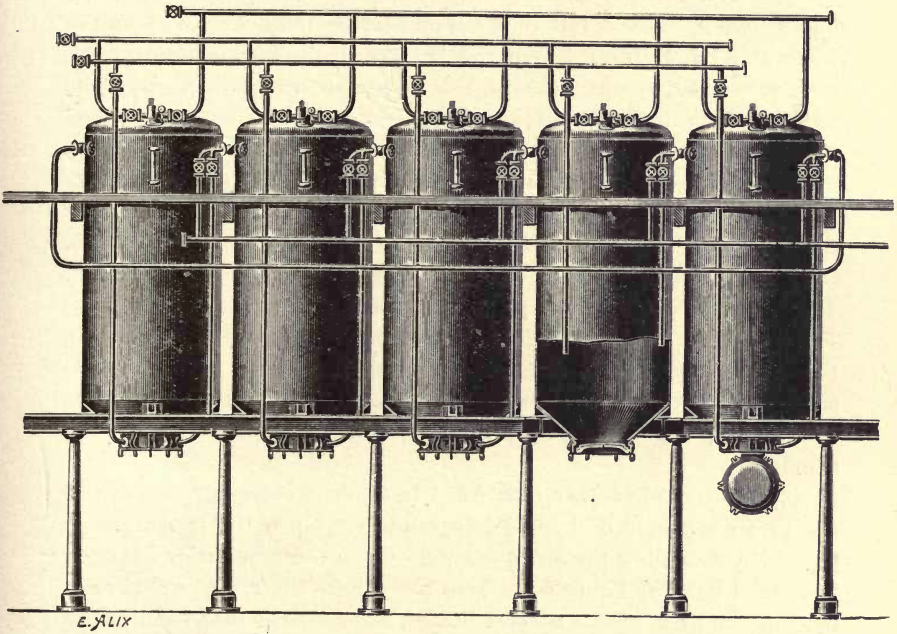

FIG. 81.—Battery of five copper autoclaves for extracting tanning substances from chestnut wood, etc.

cleaning and in charging an autoclave, there remains fifteen to twenty minutes as the actual time of decoction of the charge of chips in an autoclave. This work is intensive, and the authors in no way recommend it, because the rapidity of the passages is such that it involves a pressure which reaches 2 kilogrammes in almost all the factories which use it, and because it then fatally induces the drawbacks already mentioned. Thus, a factory treating 60 tons of bark in this way will give a yield in liquor of 3·8°, barely exceeding 1,350 litres (297 gallons) per ton of wood treated, corresponding with the loss, inherent to this method of

manufacture, to a maximum yield of 19 kilogrammes of 25° extract per 100 kilogrammes of dry wood (40 to 45 per cent. water).

Routine to Follow in Managing the Autoclaves in Intensive Working, with Five Waters giving Nine Washings.—The five autoclaves being supposed to be numbered 1 to 5 in such a way that the communicating pipes are arranged from 1 into 2, 2 into 3, etc., 5 into 1, each autoclave is successively emptied in the order of these numbers. If the series of operations be taken up at the moment when one of the autoclaves is being discharged, No. 2 for example, the following is the condition in which the extractors should be found: No. 1 full of wood and liquor from 5. No. 2 empty of both wood and liquor. No. 3 full of wood and liquor from 2. No. 4 full of wood and liquor from 3. No. 5 full of wood and liquor from 4. In the battery all the taps without exception are closed, except, perhaps, that of the steam tap of No. 3, if the water be not sufficiently hot, and if the pressure has not yet reached 1·5 kilogrammes. The following is the order in which the operations to the number of 12 succeed one another: (1) Fill 2. (2), (3), (4), (5) Pass 1 on 2; 5 on 1; 4 on 5; 3 on 4. (6) Fresh water on 3. (7) Discharge by the collector of 2 as soon as the feeding of 3 is finished. (8), (9), (10), (11) Pass 1 on 2; 5 on 1; 4 on 5; 3 on 4. (12) Empty 3; fill 3. These twelve operations are performed thus:—

(1) *Filling Extractor No.* 2.—Close the lower manhole. Feed in the wood by the top manhole. During this time apply a little steam to pack the chips, turn off the steam when it escapes with force from the top manhole. Finish filling the extractor and close the top manhole.

(2) *Running the Liquor from No.* 1 *on to No.* 2.—In extractor No. 1 the pressure should be 1 to 1½ kilogrammes. Open the steam escape tap of No. 2 to allow the air to escape. Open communication between No. 1 and 2. The liquor flows from No. 1 into No. 2, its level rises in the pipe and reaches its normal height, which shows that the transfer is finished. It is necessary to complete the amount of liquor by borrowing from 5 to balance the suction produced by the new wood. Close the communication tap. Close the escape. Apply heat by turning on steam, the pressure rises, and when it reaches 1½ kilogrammes = 127° C. (260·6 F.), turn off steam.

(3) *Passing Liquor from* 5 *on* 1.—This is done whilst No. 2 is being heated. It is carried out in the same way as No. 1 on No. 2.

(4) *and* (5) *Passing Liquor from* 4 *on* 5 *and of* 3 *on* 4.—As above.

(6) *Feed in No.* 3 *with Fresh Water.*—Communication tap closed. Steam tap closed. Escape valve open. Water tap open. Fill with water to 10 cm. below the top of the water-level and close the water

supply tap. Apply heat by turning on steam and raise the pressure to $1\frac{1}{2}$ kilogrammes = 127° C. (260·6° F.). Turn off steam at the moment that pressure is reached.

(7) *Discharge by the Collector of Extractor No. 2.*—This operation is done as soon as the feeding with water of No. 3 is finished. In this extractor the pressure ought to be 1 to 1·5 kilogrammes. Open the communication tap on the collector. The juice is propelled into the liquor vat. Close the communication tap.

(8), (9), (10), (11) *Passage of No. 1 on No. 2, No. 5 on No. 1, etc.*—These passages are effected as above.

(12) *Emptying of Extractor No. 3.*—Its liquor has just been passed into No. 4. The communication tap is closed as in all the others. The pressure is still 0·5 kilogramme. Purge the lid of the discharged manhole by the tap. Completely open the steam escape. The pressure falls, and when it is at 0 open the top manhole, then open the discharge manhole. Strip and discharge the wood. Open the upper tap even before the pressure reaches 0; the steam remaining in the extractor facilitates the opening. All the operations of charging and discharging should not take more than twenty-five minutes with experienced men (with extractors of 10 cubic metres capacity). The starting-point being again reached it is the turn of No. 4 to be discharged, and the extractors are in the following condition: No. 1 full of liquor from No. 5. No. 2 full of liquor from No. 1. No. 3 empty of wood and liquor about to be charged. No. 4 full of wood and liquor from No. 3. The liquor is about to be passed again and it will be filled with fresh water before being discharged. No. 5 full of liquor from No. 4. Operations recommence in same order changing by one unit the order of the vats, the fresh water always being run on to the vat next to be discharged. The liquor which has just been passed over fresh wood is sent to the collector, *i.e.*, to the liquor reservoirs. This is the liquor which titrates 3° to 5° Beaumé. To pass the liquor the condensation tap is opened and also the escape of the extractor which receives it, the communication and the escape are closed. Steam is turned on and turned off when the pressure reaches 1·5 kilogrammes, not to be again opened until the next passage. The liquor is thus heated each time it changes extractors.

Intensive Working with Seven Liquors.—In this case it will suffice if the battery be fitted with two collectors: the water, common to the juice, and the steam, so that the normal routine of working be that indicated by the following table:—

TABLE XIX.—SCHEDULE SHOWING ROUTINE WORKING OF EACH CELL IN DIFFUSION BATTERY DURING PROGRESS OF TANNIC ACID EXTRACTION

Routine of Working.			Duration.	No. of Autoclave.	Titre of the Liquor.
		Water on No. 7	...	...	...
Vol.		Passed	...	...	...
	7	Under pressure	...	4	...
Titre		To pass on No. 6	...	...	...
		Passed	...	...	...
Vol.		No. 6 under pressure	...	...	...
	6	To pass on No. 5	...	5	...
Titre		Passed	...	...	...
Vol.		No. 5 under pressure	...	...	...
	5	To pass on No. 4	...	1	...
Titre		Passed	...	...	...
Vol.		No. 4 under pressure	...	...	...
	4	To pass on No. 4	...	2	...
Titre		Passed	...	...	...
Vol.		No. 4 under pressure	...	...	...
	3	To pass on 3	...	3	...
Titre		Passed	...	...	...
Vol.		No. 3 under pressure	...	...	...
	2	To pass on No. 1	...	4	...
Titre		Passed	...	...	...
Vol.		No. 1 under pressure	...	...	...
	1	To be elevated	...	5	...
Titre		Elevated	...	...	...

By adopting the method of working given in the preceding table, it will be seen that the relation existing at the moment of the charge of the new wood is 2, 4, 6 figures, representing the second, fourth and sixth boiling, waiting until the first boiling be on new wood. At that moment the relation is 1, 3, 5, 7 figures, representing first, third, fifth and seventh boiling, the last which precedes the discharge of the chips from the autoclave, which have been exhausted seven times. Working thus, any number of boilings may be made, but the defect of superheating always occurs at the moment of the passages (transfers), and the pressure registered by the manometre reaches $1\frac{1}{2}$ kilogrammes = 260·6° F. With this method of working, and if a reasonable number of autoclaves be stripped in twenty-four hours, whilst exhausting to one-tenth, the tannin contained in the wood, 22 to 23 per cent. yield, is easily obtained with dry wood (40 to 45 per cent. water), corresponding to 1,650 litres (363 gallons) of liquor of 3·8° per ton of wood treated.

TABLE XX.—SHOWING EXHAUSTION OF CHESTNUT CHIPS BY CONSECUTIVE WASHINGS

Liquor.	A. Tannin per cent. in different Liquors passing successively through Chestnut Wood Chips.	B.[1] Of 100 parts of Tannin dissolved the different boilings remove the following.
1	3·8	43·37
2	2·05	23·44
3	1·23	14·04
4	0·83	9·47
5	0·42	4·79
6	0·35	3·99
7	0·08	0·90

Hence seven washings suffice. Supplementary washing continued on the seventh liquor, eighth liquor 0·003 per cent., ninth liquor 0·02, which shows that working with eight waters, and *a fortiori* nine, is useless in commercial extraction.

The exhausted chestnut chips, as they come from the autoclaves, contain 65 to 66 per cent. of moisture, that is to say that the chestnut wood treated with 50 per cent. of moisture absorbs, by maceration, 15 to 16 per cent. of water; that is the percentage that they are burned with in the gazogene (gas generator) furnace, to the total exclusion of any other fuel, for the production of the whole of the steam for an extract factory. Certain factories have tried to dry by means of a cylindrical mechanical drier, the principle of which is that of the automatic stage furnace for pyrites, utilising the heat lost by the furnaces, which removes from the wood about 40 per cent. of water; this system being adopted, the perpetual motive power absorbed (12 h.-p.) and the initial expense, rather costly, 20,000 francs (£800), do not give sufficient advantages in the calories recuperated by the removal of the 40 per cent. of water contained in the exhausted chips, seeing that simple gazogene (gas generator) furnaces are in existence, requiring no motive power nor great maintenance expenses, utilising this bad fuel in its ordinary wet condition, and yielding 1 to 4 kilogrammes of steam per kilogramme of chips burnt. On the other hand, industrial tests made for the utilisation of such wood waste for the manufacture of acetic acid and methyl alcohol have not given and cannot give any good results, because these chips must perforce be dried and yield a lower result than ordinary wood when distilled into acetic acid and methyl alcohol. Those who erected plant after the

[1] Industrial experiments made on mixtures of equal parts of barked Dauphiné, Vivarais and Lyonnais wood and on important lots of Gard wood. [It is doubtful whether the results in A were made on chestnut wood from the same sources as B, or from any specified source.—TR.]

patents of Treber, Trocknung or Fischer know in what estimation they hold the industrial value of the said processes to-day; the authors have, likewise, made numerous industrial experiments on the utilisation of this wood waste for the manufacture of paper, without any of them yielding an appreciable result.

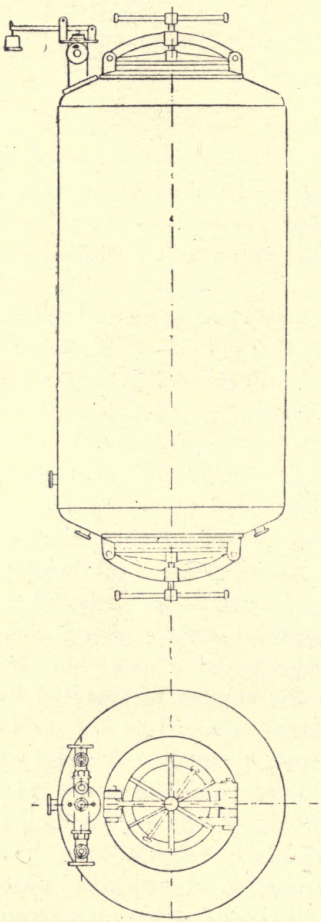

FIG. 82.—Autoclave with stamped bottom.

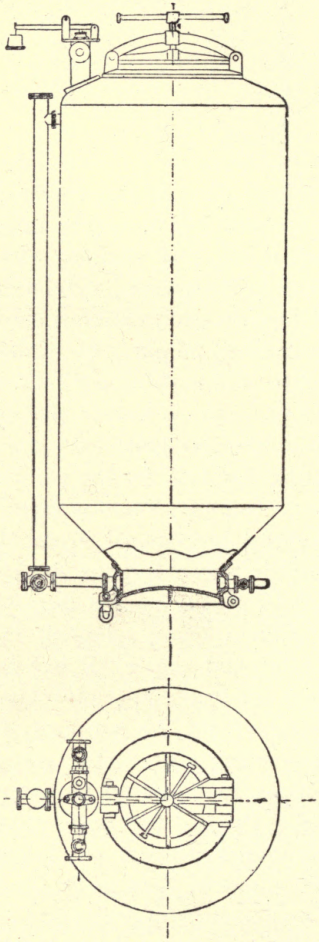

FIG. 83.—Autoclave with truncated bottom and Bonnet Spazin's arranged.

Different Systems of Autoclaves.—Figs. 82 and 83 show the different forms of autoclaves in use in tannin factories; that of Fig. 83 is the most generally employed, because it presents with its truncated base and its circular-fringed collarette in bronze, with manhole of 800 mm.

diameter, useful advantages, which the authors themselves have confirmed in a factory of which they were the advisers. These various apparatus are evidently constructed all in bronze and copper as regards the parts in contact with the juice, the armatures, the hinges and the support for closing the manholes being of steel or wrought iron.

Condensing Reheating Reservoir, J. Noyer's System.—As to the successive escapes of steam from the autoclaves at the moment of trans-

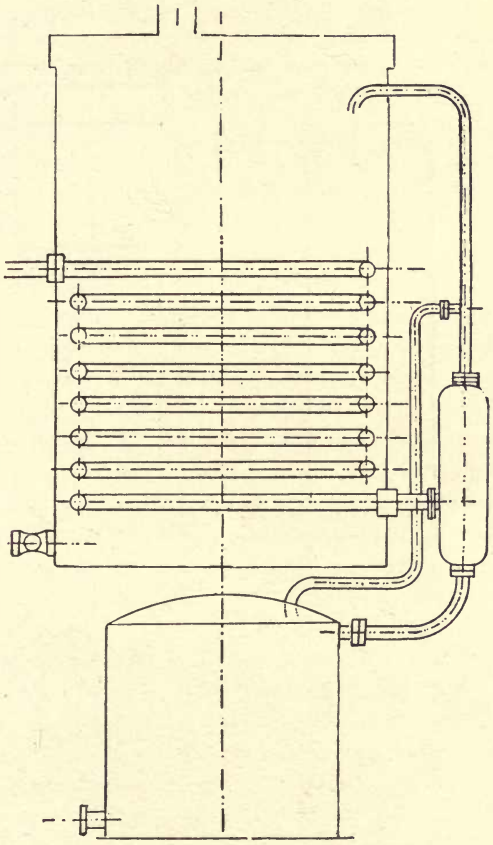

Fig. 84.—Reservoir condenser with coil, old system.

ference, it is advisable to condense this steam, so as to return to the maceration the whole amount of condensed water. For the old system (Fig. 84), which consisted in passing this escape steam through a coil, immersed in the tank, which fed the autoclaves, the authors have installed their system shown in Fig. 85. It has been designed thus, because industrially and in this special case condensation by a

worm is materially imperfect, seeing that that steam at 1·5 kilogrammes is forced to condense itself rapidly through a coil continually immersed in hot water, the spirals of which are always more or less encrusted, and, therefore, where an enormous surface of coil is required, which in the case of a 60 ton factory would have more than 21 square metres (some constructors count 0·35 square metre per ton of wood treated, which is quite insufficient), and still not enough, be-

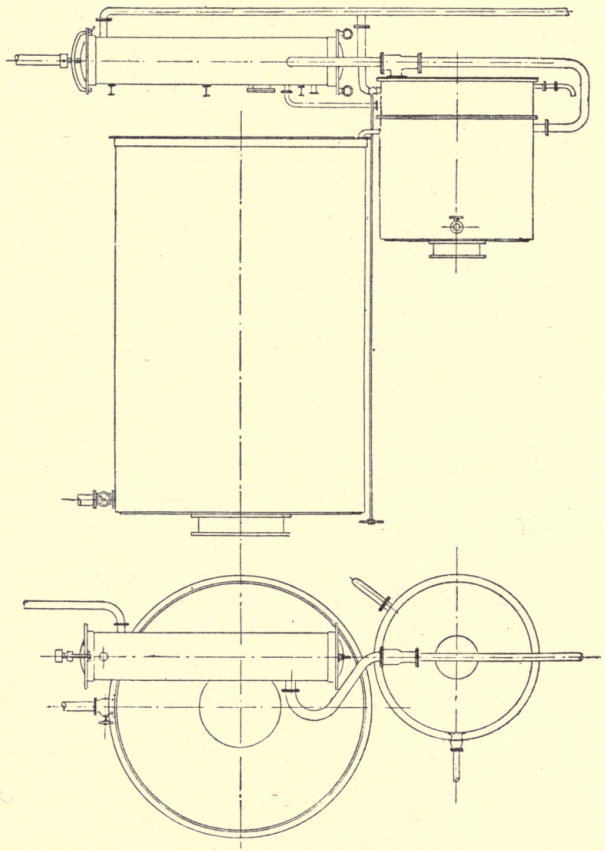

Fig. 85.—Reservoir-condenser-reheater (Noyer).

cause 10 per cent. of the steam escapes through the roof in sheer loss (without taking into account the damage produced by such projection, which is always tannic). Moreover, the surface, owing to the amount of steam to be condensed, requires such a quantity of cold water that it cannot all be utilised. On the other hand, the escape of this steam through a coil occurs by leaps and bounds, and wears it through rather

rapidly; it also requires frequent cleaning if it be desired to retain its maximum condensing surface; thus it is that for many reasons the authors have established and applied in three French factories their system of condensing-reheating-reservoir, the use of which has given full satisfaction. The plant (shown in Fig. 85) consists of a tubular reheater, the surface of which is based on the size of the extract factory, but which should not, in any case, exceed 10 square metres. It is, moreover, fitted with branch pipes, which connect it, on the one hand, with the escape collector of the autoclaves, on the other hand, with the condenser-reservoir properly so called, consisting of a copper beck with calandria, on which an emulsifier of the author's construction is fixed, the whole about 1,000 litres (220 gallons) capacity. This plant, so fitted up, is placed at the upper level of the feed tank, into which the condensed water runs. To show the advantage it realises, it may be stated that in addition to the water, which it heats from 95° to 98° C. (203° to 208·4° F.) (without taking into account that utilisable (122° to 131° F.), which not having been in contact with the tannic steam may be used to feed the boilers), it condenses almost entirely the whole of the escape steam from a battery of autoclaves of 60 tons capacity, the condensed water from which amounts to 12,000 to 14,000 litres (2,640 to 3,080 gallons), which again re-enter into the maceration work with a complement of ordinary water which exactly serves for the proper condensation, by emulsion, of the escape vapours, without fear of counterpressure or of the return of water to the autoclaves. All the pipes and manholes can be dismantled easily and readily for inspection or cleaning, without any stoppage of the factory, so as to keep the condensation capacity of the plant constant.

Liquor Tank.—Whether a battery of wooden vats or autoclaves be used, the chestnut juice or liquors are propelled from these vessels, through a special collector, to a copper tank called the "liquor tank," the useful capacity of which fully represents the volume of a vat or an autoclave, and the bottom of which is placed about the upper level of the batteries so that the juice or liquor flows to the refrigerators by gravity.

Cooling of the Juices or Liquors.—An important point in the manufacture of extracts is the cooling of the juice or liquors (the temperature of the cooled juice should never exceed 18° C. = 64·4° F.). Some factories do not use enough water because it is often badly utilised. On the other hand, its temperature often exceeds 18° C. (64·4° F.), the standard adopted by the authors, which is a satisfactory one, as the liquors can always be cooled to about this temperature; it enables the con-

densed water to be utilised for the condensation of the steam from the evaporating plant. As the amount of cold water fit for condensing the steam from the evaporation plant is always appreciably one and a half times greater than that required for the total refrigeration of the juice, it follows that the temperature of these juices approaches that of the initial water, because they traverse the refrigerators before being used for such condensation. The important point is to keep in a thoroughly clean condition the tubular bundles forming the refrigeration system, the best position for which is the vertical; the upper rim ought to be about 500 mm. above the highest level of the extraction vats so that all pumping is suppressed, every facility, however, no matter at what moment, being safeguarded for cleaning any of the organs composing the refrigerating system shown in Fig. 86.

Tubular Refrigerators.—As this treatise must be kept within a practical scope, explaining the latest and most improved systems in use, it is unnecessary to describe obsolete refrigerators, with vertical or horizontal coils, often immersed in a tank, in which the condensing water circulated, or in a channel serving the factory, because such a vertical or horizontal condition is always incompatible with frequent cleaning or control, consequently the practical impossibility of obtaining good results, seeing that the deposits of mud and encrustations formed inside and outside of the tubes forming the serpentine (which nothing can clean when at work, any more than the steam which is passed through on stopping) lower its capacity by 50 per cent.; hence non-utilisation of the condensing surface.

Only the refrigerating plant which appears up to now to have given the best results (see Fig. 86) as much from the point of view of refrigerating capacity, under the smallest volume, as that of easy cleansing of the tubular bundles of which it is composed will be described. As the drawing shows, these refrigerators are vertical and consist of five elements with tubular bundles, the interior condensing surface of which is calculated according to the quantity of juice to be cooled from 100° C. to *n* degrees (*n* being the temperature of the cold water used by the factory). For normal refrigeration, *i.e.*, under the above-mentioned conditions, and to cool the liquors from 100° C. (212° F.) to 18° or 20° C. (64·4° or 68° F.), supposing cold water at that temperature can be had, and according to the authors' experience, 1·2 to 1·5 square metres of condensing surface is required per ton of liquor at 100° C. (212° F.). The following are the differences in temperature observed on a liquor passing through five elements after issuing from the liquor vat where its temperature is 100° C.: At the first condenser 92° C. (197·6° F.), at the second 40° C. (104° F.), at the third 27° C. (80·6° F.), at the fourth

19° C. (66·2° F.), at the fifth 15° C. (59° F.). The water which was used for this refrigeration was at 15° C. and was naturally utilised for the condensation of the steam of a triple effect of 150 square metres.

Filtration of Juice Decantation.—The cooled juices, as they come from the refrigerators and according to the quality of chestnut extract to be manufactured, are filtered directly through filter presses, shown in Fig. 87, or by decantation through wooden or copper vats, the

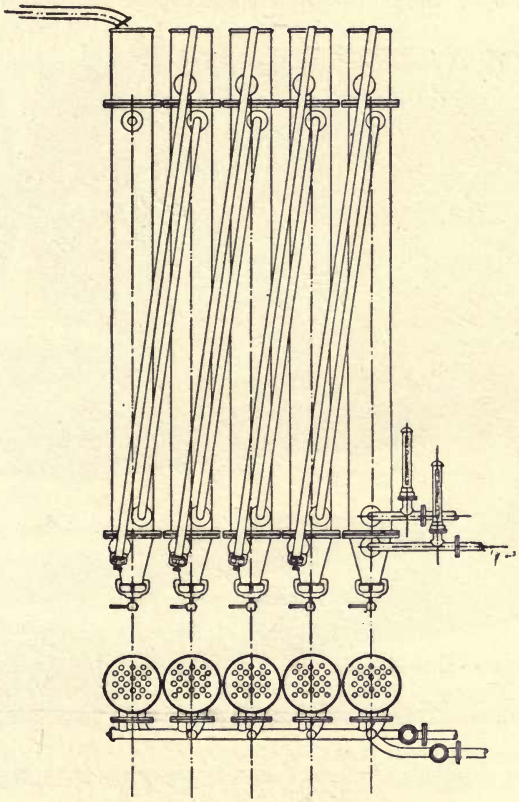

Fig. 86.—Tubular refrigerators.

capacity of which reaches 200 to 500 hectolitres (4,400 to 100,000 gallons). The filter presses which suit best in this special case are those with chambers with interposed wooden frames 800 mm. square (say 31½ inches), thirteen chambers and twelve interposed frames. A cotton cloth, of a special texture, separates the chamber from the frame, and represents about 73 square decimetres of filtering surface, say 10 square metres, for a filter of this nature. On an industrial

scale, and in observing the working manipulations incidental to the management of such plant and the washing of their cloths, their output is 110 litres (22 gallons) of clear filtrate per hour and per square metre of filtering surface, starting with crude juice, cooled to about 16° C. (60·8° F.), even taking into account the stoppages and startings after the cleanings, which are required (according to the quality of the wood treated) every four or five hours. By this process pasty cakes are formed, which fill the interposed space; they consist of wood

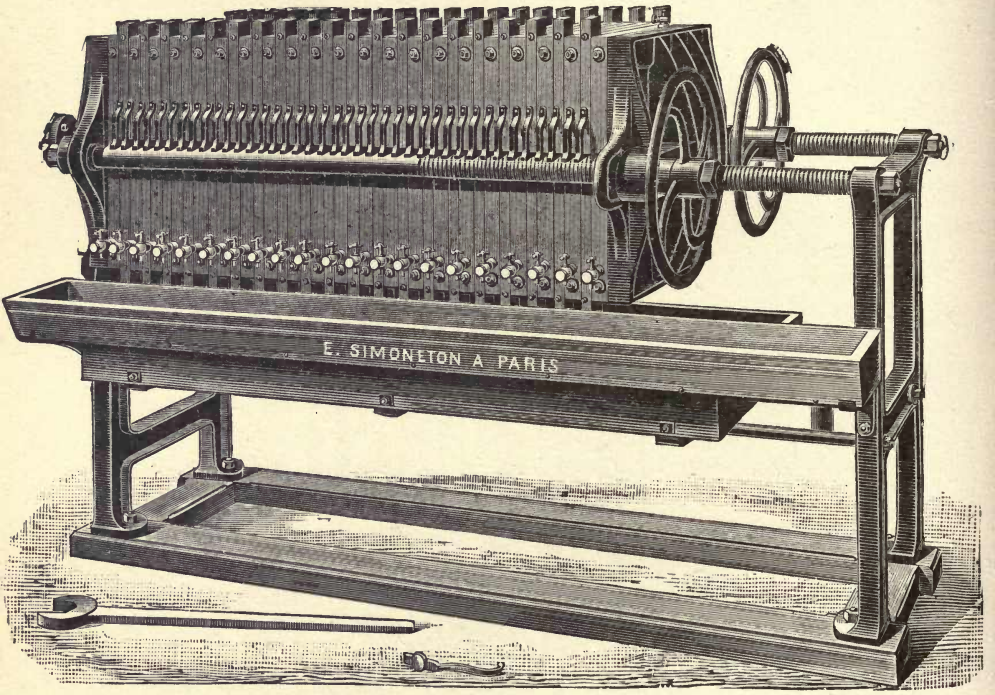

Fig. 87.—Filter press with interposed frames for the mechanical filtration of tannic liquors.

dust, mechanically entrained, and of pectosic and resinoid bodies. The juice or liquors thus treated are freed from a large amount of insoluble matter, which enables chestnut extracts of 25° or 30°, almost soluble in cold water, to be obtained, which are perfectly satisfactory to the tanner. The cakes, whether pasty or consistent, containing 6 to 7 per cent. of tannin, are again treated in a vat *ad hoc* by hot water (open steam coil); they again yield liquors of 4° to 5° B., which, concentrated to 20°, yield gallic acid for dyeing. The juice or liquors

leave 1 to 1½ per cent. of cake, which, treated as above, yield 20 per cent. of their weight of gallic acid at 20°, leaving at the bottom of the vat in which it is treated a blackish, semi-hard, resinous mass, which represents 20 per cent. of the cakes.

Mechanical Clarification.—Although clarified liquors yielding extracts soluble in the cold are obtainable by filtration, it is, none the less, a fact that this method of clarification is rather costly, owing to the motor power required for the pressure pumps and the working expenses (four men for two filter presses, clarifying 50,000 litres (11,000 gallons) of liquor), and especially the wear and tear of the cloths; the authors have tried to replace such plant by a decanting centrifugal, the principle of clarification of which is based on centrifugal force. This system was already exhibited and tested by La Société Civile des Études sur la Fabrication Perfectionée des Extraits Tanniques (Brevets, No. 161, 958 and 165, 140, 1884) without giving practical results, the style of working the authors believe being but little adapted to the nature of the liquid to be clarified; moreover, the apparatus used was not continuous, hence the output was insufficient. The authors having taken up these experiments from a different point of view, they adopted the arrangement shown diagrammatically in Fig. 88. The industrial output per hour of 1,000 litres (220 gallons) starting from cooled and decolorised liquors (immediately after treatment): 600 litres (132 gallons) of clarified juice from decantation mud fully justified their expectations.

From the above results, the authors are assured that this interesting problem is practically solved, and for this purpose they used the decanting centrifugal of Robatel, Buffaud & Co., the Lyons constructors who have built up a reputation for this sort of speciality, which the authors have been very pleased to utilise for the clarification of tannic liquors, by using their special arrangement, by which alone this object can be obtained.

Decantation in Vats.—This method of clarification in vats, which is still in general use at the present time, consists simply in collecting the cooled liquors, whether decolorised by nitrate of lead, or by blood, or by some other process, in large vessels containing 200 to 500 hectolitres (4,400 to 11,000 gallons) capacity and allowing them to settle therein, for some days, before concentration in the evaporation plant. During the interval, which varies with the process adopted and the routine of each factory, the chestnut liquor frees itself by precipitation, or coagulation, of the colouring principles, pectosic and resinoid (without counting the tannin precipitated), which are deposited in the state of mud in the bottom of the vats, to such an extent as to amount to

one-fifth or one-sixth of the liquor decanted. Thus, by the nitrate of lead process, a vat containing 20,000 litres (4,400 gallons) of liquor of 4° B., after four or five days of decantation, will yield 4,000 litres (880 gallons) of sludge. Although a battery of vats may be arranged so as to simplify the decantation process and the filtration of its muds, this style of working will always be decidedly inferior to the mechanical filtration of the juice just described. Finally, it may be added that practically on an average 4 to 5 cubic metres of vat must be calculated on for every ton of wood treated so as to realise a normal decantation. Some extract factories send these muds back indefinitely to be wrought up in the maceration, etc., but this style of recovering the tannin contained therein cannot be too severely criticised. The disadvantages incidental thereto are far from being attenuated by the compensation which is supposed to be realised by this recovery: (1)

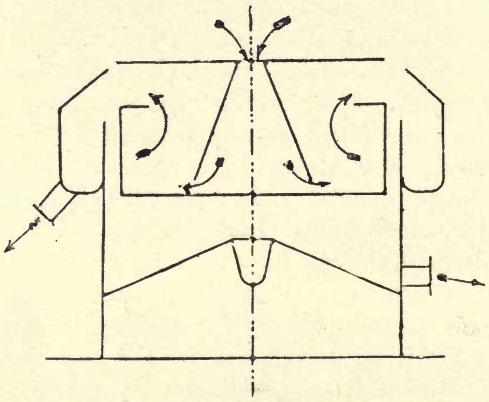

FIG. 88.—Decanting centrifugal for clarifying tannic liquors.

Because the extract corresponding thereto so produced escapes control. (2) Because these sludges (real residues) contain too many impurities to be returned to the maceration; the liquors so obtained are rendered indefinitely impure. They thus require a larger amount of decolorising agent; they are more difficult to clarify, and yield extracts of mediocre and irregular quality. (3) The piping through which the liquor circulates is rapidly encrusted and obstructed. The authors, therefore, to suppress these grave disadvantages, and to obtain the quintessence of the juice contained in the sludge, recommend mechanical filtration.[1]

[1] The authors add that for public health reasons and to avoid river pollution or the inconvenience of neighbours, tannin factories should discontinue any discharge of residues or residual waters, more or less soiled with tannin unfit for vegetation, when they do not contain organic matter from the treatment of the liquors by blood or some chemical reagent. The authors assert that in a factory using their new processes these drawbacks do not occur.

Mechanical Filtration of the Sludge.—In two extract factories which already possessed a battery of decantation vats, and to avoid the injurious and indefinite reworking up of the muds, the authors had occasion to solve this problem by mechanical filtration, using the system of filter presses described under the filtration of the crude cooled liquors. By observing the same details, a good result is inevitably realised, which reaches 25 litres (5½ gallons) of clear filtrate per hour per square metre of filtering surface. In this case the yield obtained by the filter press is four times less than in the filtration of the crude cooled liquors; that is explained if it be observed that the percentage in cakes from the mud is 10 per cent. whilst it is only 1 to 1½ from the crude liquor. Nevertheless, although this yield appears small compared with that from the crude juice, it is more appreciable, if it be considered that 100 kilogrammes of mud yield 90 kilogrammes of clear liquor and 10 kilogrammes of cake. That is, therefore, almost a maximum utilisation of these residues, which embarrassed and still embarrass certain factories. If the cakes, pasty or consistent, are not treated, as previously indicated, for the extraction of gallic acid, they are then dried in cast-iron vessels on feet on the boiler flues, so as to utilise the waste heat of the furnaces or gazogenes (gas generators) burning exhausted chips; hence economical working. These pasty cakes contain about 80 per cent. of moisture; in the dry state they only contain 5 per cent., and may be burnt in admixture with the spent chips. In a sample of cakes from filtration of the mud from juice decolorised by nitrate of lead, there were found on analysis 5½ per cent. of lead and 5 per cent. of moisture. An experiment for the complete drying of cakes already dried showed that they contained 70 per cent. of combustible or reducible matter.

Data Summarising the Foregoing Facts as to the Filtration of Liquors and the Treatment of the Cakes.—Weight of crude juice filtered per hour and per square metre of filter, 110 kilogrammes. Weight of crude mud filtered per hour and per square metre of filter, 25 kilogrammes. Cake in crude juice, 1·5 per cent. Mud in crude decolorised juice, 20 per cent. Clear liquor in mud, 90 per cent. Cake in decantation mud, 10 per cent. Moisture in pasty cakes, 80 per cent. Dry cake in pasty cake, 20 per cent. 20° gallic acid in pasty cakes, 20 per cent.

Mechanical Clarification of Sludge.—Finally, and according to the indications given above, the authors estimate that the mechanical clarification of sludges, by means of the decanting centrifugal, indicated by the diagrammatic representation in Fig. 88, is preferable to any other, and for the same reasons which apply in the case of the filtration of the juice. Moreover, the yield in clarified juice is 600 litres (132 gallons) per

hour with less labour and less wear of the cloths, because the said apparatus works by centrifugal force, so that the formation of cakes takes place on the circumference of the copper basket, and because the clarified liquor is decanted by a continuous jet by the upper portion of the same basket. The following are the advantages which accrue by adopting it in a factory treating 60 tons of wood with a volume of liquor amounting to about 90,000 litres (19,800 gallons) in twenty-four hours: (1) Four appliances of 1·200 metres in diameter of this nature will replace a decantation battery of 5,000 hectolitres (110,000 gallons). (2) The four decanting appliances only require 30 square metres of surface, whilst the decantation battery requires 300 square metres. (3) The approximate cost of four decanting appliances amounts to 16,000 francs (£640), whilst a decantation battery costs 35,000 francs (£1,400) without including the cost of construction of the building in which it is to be located. (4) The liquors being clarified as soon as cooled or treated, may be concentrated immediately; one is not, therefore, dependent on the decantation tanks, which necessitate a constant reserve and storage of the juice which is, especially in summer, essentially fermentable, an evident cause of loss in tannin.

Decolorisation. Different Processes.—The (French) patents relating to tannic acid taken out since 1879 form a long series; recourse has been made to numerous substances and chemical products.

TABLE XXI.—CHRONOLOGICAL LIST OF REAGENTS PATENTED IN FRANCE AS TANNIC LIQUOR DECOLORISERS

	Year.	Patent No.	Decolorising Agent.
1	1880	136,046	Sulphurous acid and bisulphites.
2	1879-82	130,625 / 223,951	Blood or albumen.
3	1883	155,842	Oxalic acid and alumina.
4	1883	157,153	Animal charcoal.
5	1884	161,433	Haloid or oxygenated salts with mineral or organic acids.
6	1884	155,026 cert. ad d.	Sulphurous acid gas under pressure.
7	1884	163,189	Aluminium hyposulphite.
8	1884	163,521	Barium chloride.
9	1886	174,972	Casein with prior treatment of wood by sulphuric or hydrochloric acid.
10	1886	56,304	Lead nitrate.
11	1899	290,159	Lactic acid.
12	1895-97	242,041 / 269,628	Dregs of wheat, barley, maize or rice, oil cake.

These processes will here be put to one side as quite incompatible with the manufacture of good extract, as the tannery can, and should, restrict itself to deliveries more or less in accord with its system of

tanning; extracts decolorised in so many different ways are no guarantee to the tanner using them, after frequently having had mishaps often due to them alone. In support of the foregoing, it will suffice to point out the loss in extract, and consequently in tannin, which results from the decolorisation of liquors by the "blood" process (one of the most rational) and which, in the same way as in the clarifying of wine, entrains along with the tannin the pectic, resinoid and colouring principles of the liquor; thus 40,000 kilogrammes of liquor treated with Bourgeois' solution of blood (at 6·5° B. and 7·5 kilogrammes per 1,000 kilogrammes of liquor at 4° B.), which ought practically to give 6,400 kilogrammes, only yields 5,760 kilogrammes, say a loss of 10 per cent. in extract, or 16 per cent. on the weight of the liquor, which, after treatment, only titrates 3·6°, say a fall of 0·4°. As to the "nitrate" process, it is still worse, because the formation of free nitric acid eventually destroys again a notable amount of tannin, owing to secondary reactions (in presence of this acid) in the concentration of the liquor in the evaporation plant, aided by the heat of that operation, to say nothing of the wear and tear by corrosion of the vessels in question by the presence of nitric acid after a shorter or longer lapse of time. The authors tried to neutralise this reaction by the addition of pure precipitated carbonate of lime, but the remedy was more aggravating than useful in the decoloration of the juices so treated. The contentions of the authors in regard to the decoloration of extracts are supported by the fact that numerous British and German tanners have used the crude extracts for a long time or extracts simply clarified (by one of the mechanical processes already described), which ensure uniformity in the deliveries which constitute their important markets, and especially a higher percentage of tannin, and consequently a higher yield in leather. The decolorisation of the future does not lie in the use of chemical products, nor in more or less complicated processes; tanning is an industry already far too complex to stand to it; it requires, and will require, before and above all pure extracts, of a well-determined nature, with a high percentage of tannin, soluble in cold water, and finally of as constant and uniform a composition as possible. However, Peyrusson's curious process of decolorisation, Brevet No. 318,523 (8th Feb., 1902), with certificate of addition No. 318,523,467, in which the patentee specifies that the process described in his principal patent rests especially on the action of the presence of tin, applied during the preparation of the tannic and tinctorial liquors and extracts, with the object of preventing the grey coloration of the latter. Thus in treating chestnut extract in a digester of any sort, in presence of tinfoil, it will be found that the wood remains white,

whilst the same wood treated in the same way, but without tin, assumes a grey or even a brown tint. Finally, there may be quoted as a recent patent that of George Klenk, for a process of decolorising tannic liquors by aluminium sulphate and sodium bisulphite, as follows: Add to the hot liquors from the extractors, in a vat fitted with an agitator, a solution of sulphate of alumina (the quantity to use depends on the strength of the juice); after the mixture of these two liquors, add thereto bisulphite of soda 38° to 40° B. *q.s.*, stirring constantly. The following is the reaction:—

$$\underset{\text{Aluminium sulphate}}{Al_2(SO_4)^3} + \underset{\text{and Sodium bisulphite}}{6NaHOSO_2} = \underset{\text{Aluminium hydrate,}}{Al_2(HO)^6} + \underset{\text{Sodium sulphate,}}{3Na_2SO_4} + \underset{\text{Sulphurous acid.}}{6SO_2}$$

The average proportions to use are liquor 4°, 5,000 litres (1,100 gallons), solid aluminium sulphate 4 kilogrammes (8·8 lb.), sodium bisulphite 38° to 40° B., 15 to 20 kilogrammes (33 to 44 lb.). This process is applicable to quebracho, mimosa, hemlock, sumac, pine, oak, chestnut and others. The juice so treated is decolorised by aluminium hydrate in the nascent state, which agglutinates the resinoid particles of the liquor, and falls with them to the bottom of the vat, precipitated in the same way as with albumen or blood. Moreover, the nascent sulphurous acid, which escapes during the decolorisation of the juice, has an intensive decolorising effect. After cooling to 20° to 25° C. decantation, concentration, etc., extracts are got soluble in cold water, retaining an acid reaction giving a pale yellow colour to leather, differing slightly but sometimes not at all from oak tanned leather. Finally, the colour imparted to the leather by such extracts is permanent and is not liable to change under the ordinary action of the air. Notwithstanding the interesting aspects of these decolorisation processes, the authors maintain the opinion given above, and add that they all have drawbacks, both for the extract manufacturer and the tanner; the first loses, whatever may be the process used, from 2 to 3 per cent. of tannin to the detriment of the yield in extract, and this percentage fails the second party, whilst the colour of his leathers is in no way altered. The authors can, in fact, affirm that, having had occasion to make important analyses of the tannin in different leathers, where they had used decolorised extracts (either by blood or by nitrate), all the leathers tanned by these extracts were darker than those yielded by other and well-clarified extracts, and that although the coloration of a 1 per cent. solution was less dark than a solution of clarified extract. Moreover, the tannery of the future will be that which utilises mixed processes of tanning, with extracts manufactured in a genuine manner and judiciously used; the opinion of Procter, the eminent English

chemist, is decisive on this point. If it be desired to impart to manufactured extracts the necessary degree of solubility, resource must be made to perfectly neutral materials, products already used by the tannery without ever giving rise to any drawback; thus the authors, rigorously inspired by these conditions, have used borax to render chestnut extract soluble. The borax is used in tanneries as follows: "Borax may be used for many purposes in tanning and currying; its most important rôle is to soften the river water, to clean and preserve the skins, to avoid the loss of their gelatine, and thus to render the leather heavier and firmer; it prevents the pits from going bad and putrid, it is the most efficacious and harmless agent that can be found for preliminary rinsing and cleaning of skins before being placed in the vat. With a kilogramme (2·2 lb.) 1,000 litres (220 gallons) of the water generally used may be softened. Solution is effected in boiling water and it is run into the vat with energetic stirring." It was, therefore, after important and repeated tests, that the authors were led to adopt it as a solvent agent for extracts clarified according to the mechanical processes already described; the factory where the authors, *inter alia*, demonstrated this interesting question continues to use it on an output of 7,000 kilogrammes per day, and they know, on the other hand, that the tanners who use it are perfectly satisfied therewith. At the minimum rate at which it is incorporated, it only enters into the composition of these extracts, so as to render the small percentage of insoluble, soluble in the cold. Added to the liquor, at a certain stage of the manufacture, it produces neither precipitate nor reaction of any kind.

Manufacture of Chestnut Extracts of 25° B., 30° B. or Dry. Concentration or Evaporation of Chestnut Extracts.—The treated juice or liquors, *i.e.*, clarified according to the processes already described, pass directly from a liquor receiver to the evaporating vessels, represented in some factories by a double effect, in others and better still by a triple effect, which constitutes, in the opinion of the authors, the plant which ought preferably to be used. (The different systems used in tannin manufactures will be described farther on.) As stated, it must be taken that liquors of 3·8° yield 13 to 14 per cent. of extract of 25° to 26°, with 86 to 87 per cent. of water to evaporate; knowing on the other hand that the ton of wood treated yields on an average 1,350 to 1,650 litres of liquor (297 to 363 gallons) according to the method of working adopted, it is easy from such data to determine the evaporative capacity to impart to the vessels intended for the concentration of the liquor of our typical factory, the consumption of which is 60 tons per twenty-four hours, which will be, moreover, described later on. These vessels are fed in a

regular manner, and it may be said that it is even possible to feed them automatically so as to do away with an attendant. A triple effect evaporating 20 litres of water per hour per square metre will thus yield the corresponding quantity of extract of 25°, say 2·7 kilogrammes. In No. 3 it may be taken that this hourly output will fall to 2·1 kilogrammes when making 30° extract. As to the steam consumption and always in the case of a triple effect, it is necessary to allow 7 kilogrammes for the evaporation of 20 litres of water per square metre per hour; finally, if a reheater be utilised prior to No. 1, the consumption may be appreciably reduced, and it may be said that the evaporation of that quantity of water apparently requires the amount of steam which corresponds to 1 kilogramme of coal or to 5 kilogrammes of spent chips. Although this yield appears satisfactory and suffices on normal working, the Kestner system, which gives far superior results, will be explained further on. No. 3 is generally furnished with a syrup discharger or a special extract pump by which the extracts, when they have reached 25° or 30°, can be evacuated whilst the plant is at work and stored in tanks where they cool naturally before being sent out in petroleum casks. It may be said that these extracts, once placed in the vats, gain by being left there to deposit for several days; the mud which they deposit can only spoil the quality. It is better to take these deposits and treat them together with the mud from the clarified juice; they can then be concentrated.

Dry Extract.—It is possible, by the rotary simple effect described later on, to push the concentration farther than 30° and to bring it to the dry condition (its composition has been given); it is then necessary, so as to produce 100 kilogrammes, to use 210 kilogrammes of 25° extract, or 170 kilogrammes of 30°, consuming 3 to 4 kilogrammes of steam. Its cost price is then fixed at about 25 francs (£1) the 100 kilogrammes (2 cwt.) for 45 per cent. tannin.

Solution of Dry Extract.—To dissolve dry extract, do not use too cold water, and if perfect solution be required heat to 60° C. (140° F.) or higher. Crush the cakes to be dissolved and suspend the pieces in a willow basket in the upper part of the liquor. Let solution take place of its own accord until the solid extract has disappeared. If the cake of extract be placed in the bottom of the liquid, it is impossible to dissolve a dry extract well even when stirred continually with a rod. Only extracts soluble in the cold are included in this style of working, prepared as indicated by the authors; extracts containing a percentage of insolubles (more than 5 per cent.) require to be rendered soluble in an apparatus *ad hoc*, which will be described farther on when dealing with dry quebracho extracts (see p. 260).

Factory Routine.—It is indispensable for the good working of an extract factory that it be subjected to rigorous control day and night; a manufacturing schedule is therefore appended here with its control appendix by which the manufacture may be followed daily and at no matter what moment. These documents also serve as the basis of strict book-keeping, hence errors, so to speak, become impossible without it being shown one way or another by the quarterly inventories which should be taken in a factory of this kind.

TABLE XXII.—MANUFACTURING SCHEDULE WORKS RETURN SHEET No. , DATE , 1905. OAK AND CHESTNUT EXTRACTS

Oak. Quantity.	Lot and time in shed.	Wood treated. Quality Source.	Wood treated. Quantity.	Number of vats or autoclaves.	Extract produced deducting 3 per cent. for waste. Quality.	Extract produced deducting 3 per cent. for waste. Quantity.	Decolorisation. Nitrate.	Decolorisation. Blood.	Number of workmen. Manufacture.	Number of workmen. General Expenses.	Wood consumed in lighting the furnaces.	Decantation movement of the vats. No. of Vats B.R.	B.P.
Report		day :		day :								1	
												2	
												3	
												4	
												5	
		night :		night :								6	
												7	
												8	
												9	
	Totals											10	

Sales. Extract 25° clarified soluble.	Extract 25° decolorised.	Extract 25° special.	Extract 30° clarified.	Extract 30° decolorised.	Oak 25° clarified.	Gallic acid 20° for dyeing.	Wood in Stock. Stock.	Wood in Stock. Used.	Extract in Warehouse.	Remarks.
									25° C.	
									25° D.	
									25° S.	
									30°	
										Urgent repairs.
									Oak 25° G. 20°	
										Chief works chemist.

TABLE XXIIA.—SCHEDULE ANNEXED TO TABLE XXII., No. , DATE , 1905. CONTROL

Average of the tables relating to each department and signed by the foreman or by the man in charge.

	Observations.	Repairs. Day			Night		
1	Average pressure on the boiler						
2	Average strength of the liquors						
3	Number of triple effect boilings						
4	Average temperature of the water feeding the boilers						
5	„ „ „ „ feeding the autoclaves or vats						
6	„ „ „ „ from the refrigerators						
7	„ „ „ „ from the condensers						
8	„ „ „ cooled liquors						
9	„ „ „ hot gases						
10	State of the furnaces						
11	Draught in mm. at the fan or chimney						
12	Average vacuum in the three vessels	1	2	3	1	2	3
13	„ temperature of the three vessels						
14	Weight of 25° or 30° extract made vat No.						
15	Tannin per cent. of average liquor						
A	Per cent. of tannin in 25° extract						
B	„ „ „ 30° „						
C	„ „ „ gallo-sumac						
D	„ „ „ quebracho						
E	„ „ „ special 25°						
F	„ „ „ oak 25°						
	Tannin per cent. of wood treated, Lot No.						
	Moisture „ „ „ „						

Chief works chemist.

Evaporation by Direct Steam.—Following the example given by M. P. Kienlen in his remarkable "Examination of the Different Systems of Evaporating Lyes" in his "Soda and Soap Industries" (*Mon. Scientifique,* 1898), the authors propose in their turn to pass in review the different evaporation plant used in the important tannin industry for the concentration of the juice or tannic liquors, from 4° to 25° or 30° B., the hydrometer strength at which they are valued commercially.

Concentration by Indirect Heat: Evaporating Pan with Chenailler's Coil.—As in this industry, the use of direct steam for the concentration of the juice—whether in a pan fitted with a steam coil or with a Chenailler's coil—oxidises and destroys the tannin by the too great temperature of the steam (150° C. (302° F.) with a pressure of 5 kilogrammes) passing through this style of apparatus in the open air, and as it would be necessary to give them cumbersome dimensions, and at the same time to consume an excessive amount of steam, it will only be adverted to as a matter of history, and mention will only be made of Chenailler's lens evaporating plant (Fig. 89), which might be used at a stretch for the evaporation of liquor intended for crude chestnut extracts (20° gallic

for dyeing), or logwood or other dry tinctorial extracts. It need only be added that a Chenailler's apparatus expends 25 kilogrammes of steam per hour per square metre, when a simple effect expends 1·1 kilogrammes, a double effect 0·72 kilogramme, and a triple effect 0·360 kilogramme to evaporate the same amount of water.

Plant for Evaporation under Reduced Pressure (*Vaccum Pans*).—It is to the sugar industry that we owe the first plant, based on the principle of lowering the boiling point of a liquid at the atmospheric pressure, by bringing that liquid to the state of vapour under a pressure less than that of the atmosphere. Howard, the engineer, was the first to introduce into British sugar refineries, in 1813, the first appliance of this nature, using, moreover, for the production of the vacuum a barometric condenser with countercurrent and dry air pump. Later on

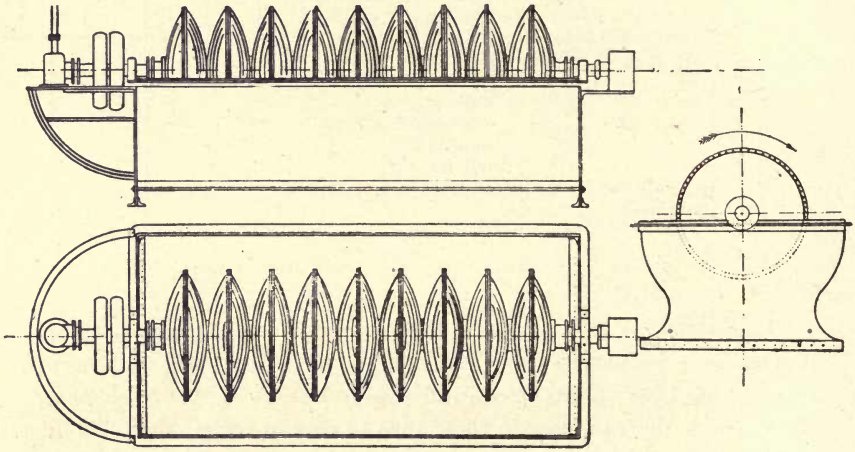

Fig. 89.—Chenailler's evaporator.

Derosne sent out analogous plant, but with surface condensers in which the diluted juice acted as the refrigerating agent. Finally, the principle of the multiple utilisation of heat was applied in America by Rillieux in 1830 then in Europe about 1850. During this time numerous improvements were brought to bear thereupon by Tischbein, Robert, Degrand, Walkhoff, Cecil (? Cail) and Derosne, but the merit of their propagation in the French sugar industry belongs to Cail & Co. and more recently to the Fives-Lille Construction Co., without taking into account the firm of Bonnet-Spazin, who have specialised in the construction of plant intended for use in the tannin industry since 1877. Such plant is constructed on the principle of utilising the latent heat which is disengaged from the liquid being evaporated (and

which is generally lost in those pans that are heated indirectly or in the open air) to heat fresh quantities of liquid to be evaporated in one or more vessels communicating consecutively with each other. The arrangements for simple, double and triple effect used in the tannin industries will be described and their maximum utilisation, as much from the point of view of the amount of steam evaporated per hour as from that of the economy in steam to be realised, all proportions being adhered to so as to ensure a high capacity.

Simple Effect.—This, the simplest type, comprises a closed pan (which the French call a *caisse*) in which the pressure is lowered by the rarefaction of the air at the exit of the steam from that pan. The heating steam (the escape steam from the engines or coming directly from the steam boilers) is injected into the steam chamber of the pan. The calandria is provided with two horizontal tubular bronze plates pierced with holes, in which there are fixed *par dudgeonnage* the tubes of a tubular bundle, thus forming a large heating surface. There is formed in this way around this tubular bundle a heating chamber occupied by steam, the juice to be evaporated being inside the tubes. The authors remark that it would be more normal to cause the steam to pass inside the tubes, after the style of a tubular reheater, and to cause the juice to be evaporated to circulate all around the exterior; however, practice has shown that the converse should be the case, looking to the facility for cleaning the tubes which, eventually becoming encrusted, require cleaning by brushes, so as to preserve the maximum evaporating capacity of a tubular bundle. The steam penetrating into the heating chamber condenses, abandoning its latent heat to the juice to be evaporated, the latter boils under the reduced pressure, and thus yields a fresh amount of steam. The water from the condensation of the steam in the heating chamber is run off through a blow-off pipe, and generally by an automatic blow off which prevents the passage of steam. It is used to feed the boilers. The steam from the boiling juice is condensed in a surface condenser, placed in front of the air pump, at the exit from the pan, and yields a corresponding quantity of hot water, which is used for macerating the wood. With a barometric or with an injection condenser, the steam mixed with the condensation water goes down the drain in sheer loss, therefore the authors recommend the exclusive use of a surface condenser in the three cases, of simple, double and triple effect, because they hold, as seen previously (Chap. I.), that in a tannin factory the maximum yield and highest percentage in tannin are obtained by the use of water as pure as possible for the maceration of the wood. The condensed waters from the evaporating vessels

are obviously indicated, and, in default of a system of surface condensation to collect them, a special pump is required called the condensed water pump, whether in double or triple-effect working.

Installing a Simple Effect.—This apparatus, now slightly modified, was described in its simplicity by Othon Petit (*Des Emplois chimiques du Bois dans les Arts et l'Industrie,* 1888). A more improved form of this plant is, therefore, shown here (Fig. 90), combined with its juice reheater, R, and a wet air pump. That arrangement is particularly suitable for tanneries of a certain magnitude, which wish to concentrate

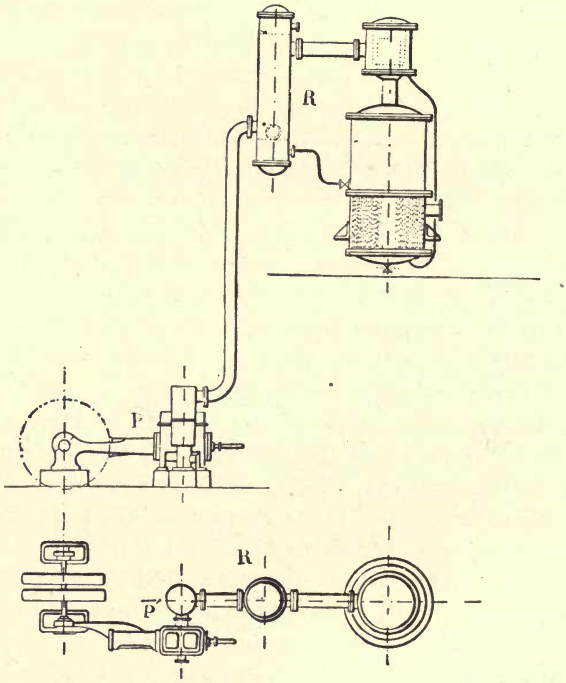

FIG. 90.—Simple effect evaporation plant with reheater and its pump.

bark, diffusion juices, or juices from the maceration of wood, or weak juices regenerated from their train of vats, after treatment and clarification, manufacturing in this way easily and economically thick juice of 12° to 15° B. which serves to enrich those at work. Here are the details of this plant which comprise: (1) A vessel, A, of 20 square metres of heating surface, capable of evaporating 1,000 litres (220 gallons) of water per hour. This apparatus is made entirely of copper and bronze in those parts which come into direct contact with

the juice and consists of a tubular calandria 1 metre in diameter, height between the tubular plates 0·77 metre, thickness 5 mm., in the bottom 6 mm.; an upper calandria, diameter 1 metre, height 1 metre, thickness 5 mm., in the bottom 6 mm.; a safety vessel, V, 600 by 600 mm., thickness 3 mm., in bottom 4 mm.; two tubular bronze plates, 16 mm. thick; a tubular bundle comprising 162 tubes in electro-copper of 50 mm. of exterior diameter, thickness 2 mm., a copper steam inlet connecting pipe on the tubular bundle, a copper connecting pipe for the passage of the steam to the safety vessel, a connecting pipe and a pipe of 35 mm. diameter for the return of the entrained liquor; iron joint bands; cast-iron supports. It is also fitted with its taps and the following accessories: one bronze tap 40 mm. for liquor inlet. One bronze three-way tap 10 mm. for discharging the extract and the wash water. One connecting pipe all bronze for blowing off the condensed water. Gauge glass vacuum indicator. Air tap. Anti-froth tap (*robinet à beurre*). Wash water tap. Test glass for sampling. Manhole doors in bronze 400 mm. in diameter. Round peepholes 100 mm. Tap for evacuation of incondensable gases. The complete plant weighs 1,625 kilogrammes (say $1\frac{5}{8}$ metric tons or about 32 cwt.). The air pump, P, serving this triple effect, with its condenser, C, entirely of copper and bronze in the parts connected with the tannic steam, capable of condensing 100 kilogrammes of steam per hour, is driven at will by a belt, or by a direct motor.

Juice Reheater.—As has been remarked, and as is, moreover, shown in Fig. 90, the juice to be concentrated traverses the tubular bundle of a copper reheater (Fig. 91), consisting of a cylindrical calandria of 2 metres by 400 mm., of two tubular bronze plates, one upper manhole, one lower manhole. The tubular bundle consists of thirty-seven copper tubes of 40 × 45 mm., representing 8 square metres of heating surface. A juice inlet connection pipe for the distribution chamber and a discharge pipe complete this apparatus, into which the juice runs, between 15° to 20° C. (59° to 68° F.), to issue between 50° and 55° C., a temperature at which it is brought by the steam from the simple effect circulating round the tubular bundle of the reheater before it passes to the air pump and the condenser. This simple apparatus realises a notable economy in steam, which may be estimated at 9-4, 5-3 per cent. in triple, double or simple effect.

Rotary Simple Effect.—The rotary type of simple effect, designed by Bonnet-Spazin & Co., of Lyons, in which the tubular bundle revolves, may be described here. This system, where the juice to be concentrated circulates around steam pipes, is especially suited for the manufacture of chestnut quebracho, logwood or other dry extracts. It

advantageously replaces Chenailler's system previously described. It consists (Fig. 92) essentially of a horizontal copper calandria, fitted with a double bottom, and surmounted by a safety vessel, inside of which is a froth breaker, two bottoms or linings of copper plates pierced with a central hole closing its two extremities.

Inside a tubular bundle, consisting of copper pipes 2 mm. thick, 40 by

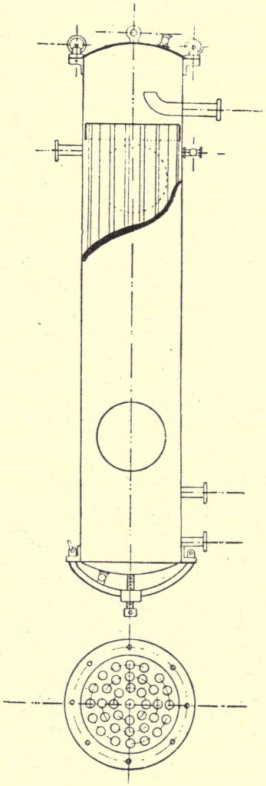

Fig. 91.—Tannic liquor reheater.

45 cm., the extremities of which are dudgeoned into the orifices of two tubular bronze plates of 18 to 20 mm. thick. A hollow central shaft traverses the whole system from one end to the other, and receives the steam, which it distributes into all the pipes, and enters the tubular plates and the linings of the tubular bundle, where the condensed steam is blown off through the double bottom to be afterwards evacuated. The tubular bundle turns on the glands of two stuffing

boxes, which are fixed in the centre of the dome part of the calandria ; the extremity of the hollow shaft again rests on two bearings with stuffing boxes with glands, the one serving as a steam inlet, the other as an outlet for the condensed water evacuated by the rotation of the tubular bundle and the pressure of the steam, which cause it to pass into the double jacket, whence it escapes to be utilised in feeding the boilers.

Double Effect.—This type consists of two closed vertical pans communicating with each other, in which the pressure is lowered by rarefaction of the air at the extremity of the system. The heating steam, as in the simple effect, entering the heating chamber of the first pan, condenses, giving up its latent heat to the liquid to be evaporated, the latter boils under slightly reduced pressure, and thus yields a fresh quantity of steam. This steam is utilised to heat the tubular bundle of No. 2 vessel communicating with No. 1 ; it causes fresh liquid to

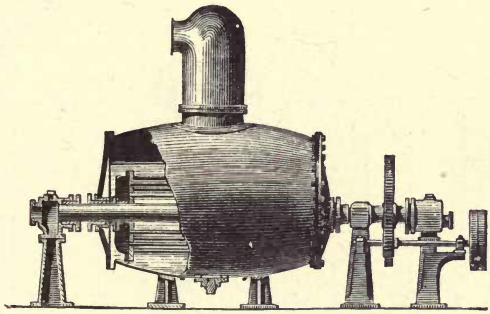

Fig. 92.—Horizontal rotary simple effect concentration plant.

boil under the reduced pressure, produced by the rarefaction of the air in the last vessel, by means of a special pump. Fresh quantities of water condense in the heating chamber of No. 2 ; the steam arising from the boiling of the liquor in that vessel is condensed in a surface or injection condenser placed in front of the air pump at the exit of No. 2, after having traversed the tubular bundle of a juice reheater. The heating chamber of No. 2 may be connected with a condensed water pump (the water from which is used for the maceration of the wood), a centrifugal pump, for example, which pumps it to the condensed water reservoir feeding the vats or autoclaves. Fig. 93 shows a double effect with 90 square metres of heating surface, capable of evaporating 2,250 litres (475 gallons) of water per hour with barometric condenser, condensed water pump and dry air pump wrought tandem by direct motor. The safety vessel shown is of the system with multiple perforated baffles ; it gives very good results, as a froth breaker,

in the evaporation of oak liquors, which froth greatly. It may be remarked, that an increase in the number of vessels does not result, as one might believe, in an increase in the production of the system, but simply in a decrease in the steam expended, as also in the amount of water used for condensation. Moreover, the number of vessels appropriate for the work does not depend so much on certain peculiar cases, as in alkali manufacture, or in sugar manufacture; the authors'

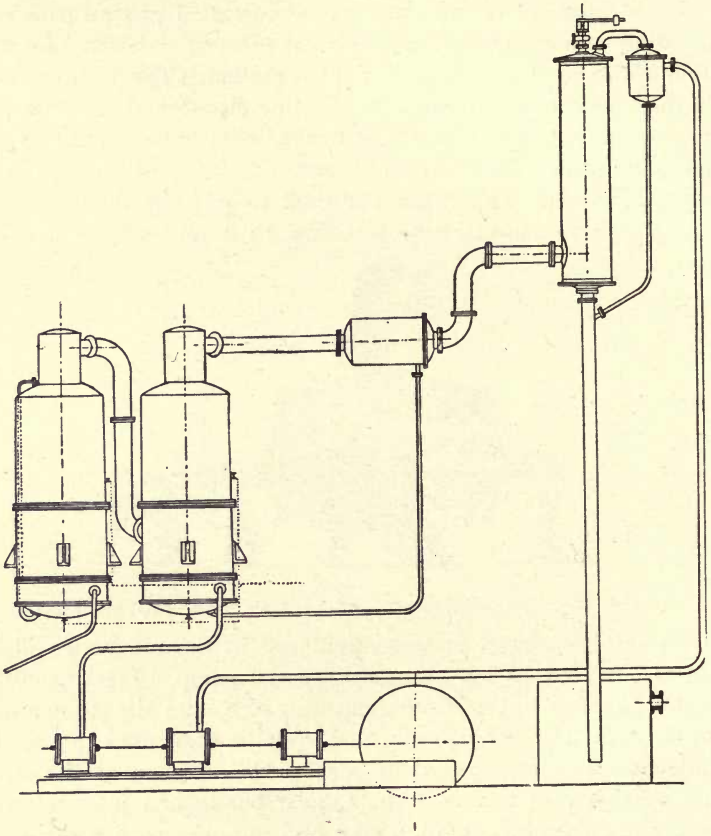

FIG. 93.—"Double effect" with its barometric condenser and its two pumps driven in tandem by direct motor.

experience in the matter enables them to assert that for a tannin factory to work economically, that is to say, to use exhausted wooden chips solely as fuel (the use of coal would, moreover, point to abnormal working, bad arrangement of plant and apparatus, or obsolete processes), it suffices to use an improved triple effect.

Theory of the Triple Effect.—Suppose the heating steam in No. 1

be 100° C. and the liquid arriving at its boiling point, say, 84° (see Fig. 94). The heating steam is condensed at 84° C.; it gives up a quantity of heat: latent heat 84°, $496{\cdot}3 + 40{\cdot}200 + 16 = 552{\cdot}50$ calories per kilogramme of steam. The liquor in No. 1 is vaporised at the temperature of 84° C. To vaporise a kilogramme of water at that temperature an amount of heat is required of $507{\cdot}12 + 39{\cdot}04 = 546{\cdot}16$, hence 1 kilogramme of condensed steam in No. 1 vessel can vaporise $\frac{552{\cdot}50}{546{\cdot}16} = 1{\cdot}015$ kilogrammes of liquid in that vessel. No. 1 steam at 84° condenses in No. 2 at 73° C. Each kilogramme of that steam, converted into water at 73° C., gives up $546{\cdot}16 + 11 = 557{\cdot}16$ calories. The liquor entering No. 2 at a temperature of 84° C. is converted into

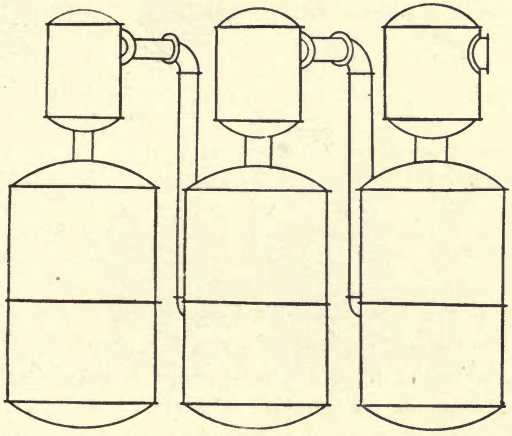

FIG. 94.—No. 1. Vacuum 35-40 cm. No. 2. 50-53 cm. No. 3. 60-67 cm.

steam at 73° C.; it requires for that an amount of heat of $546{\cdot}16 - 11 = 535{\cdot}16$ calories. Hence 1 kilogramme of steam condensed in No. 2 vaporises $\frac{557}{535} = 1{\cdot}039$ kilogrammes of liquid in that vessel. No. 2 steam produced at 73° C. is condensed in No. 3 into water at 54° C. One kilogramme of that steam at 73° C., converted into water at 54° C., gives up a heat of $550{\cdot}6 + 19 = 569{\cdot}6$ calories. The liquor entering No. 3 is at 73° C. and it is reduced to steam at 54° C. It requires for that per kilogramme a heat of $550{\cdot}6 - 19 = 531{\cdot}6$ calories. Hence a kilogramme of steam condensed in No. 3 vaporises $\frac{569{\cdot}6}{530{\cdot}6} = 1{\cdot}071$ kilogrammes of water therein. To sum up, 1 kilogramme of steam entering No. 1 at 100° C. produces a kilogramme of condensed water at 84° C.; 1·015 kilogrammes of steam in No. 1, which is converted into 1·015 kilogrammes

of condensed water at 73° C., will produce 1·015 × 1·030 = 1·050 kilogrammes of steam in No. 2, which condense into 1·050 kilogrammes at 54° C., which produce 1·050 × 1·070 = 1·125 kilogrammes of steam in No. 3, which goes to the condenser. Hence, finally, 1 kilogramme of steam at 100° C. induces the evaporation of 1·015 of water in No. 1, of 1·050 in No. 2, of 1·125 in No. 3—a total of 3·190.

TABLE XXIII.—SHOWING BALANCE OF THE ACCOUNT OF STEAM EXPENDITURE AND RECOVERY IN THE WORKING OF TRIPLE EFFECT PLANT IN THE CONCENTRATION OF TANNIN EXTRACTS

There were in the beginning :—

	Calories.
1 kilogramme of steam at 100° C., containing	636
3·190 kilogrammes of liquor at 84° C., containing	336
Total	972

There is recovered at the end of the operation :—

	Calories.
1 kilogramme of water at 84° C., possessing	84
1·015 kilogrammes of water at 73° C., possessing	74
1·050 „ „ „ 54° C., „	56
1·125 „ of steam at 54° C., „	698
	912

Weight of Water Evaporated per Vessel ("caisse") by a Triple Effect Plant.—The evaporative capacity of a vessel is proportional to the heating surfaces and to the difference in temperature between the steam and the liquid. It depends on the state of the tubes and of the density of the liquid in virtue of its resistance to the motion of circulation. Let the two last elements be neglected. No. 1, 1 square metre; No. 2, 1 square metre; No. 3, 1 square metre. Take the temperature of the heating steam as 100° C., the vacuum as 65 cm. in No. 3 giving 54° C. Total difference, 100 – 54 = 46. Dividing this difference into three, say 15·3, the apparatus ought to be regulated to have a temperature of

100 – 15·3 = 84·67 in No. 1, 76 – 43 = 33 cm. vacuum
84·67 – 15·33 = 69·34 in No. 2, 76 – 22 = 54 „ „
69·34 – 15·34 = 54 in No. 3, 65 „ „

and the three vessels will do the same amount of work.

Triple Effect.—As already stated, the triple effect system of evaporation under reduced pressure is much the best system for tannin factories or dyewood extract factories. It ensures constant and economical working by its comparatively great evaporating capacity, coupled with a minimum steam consumption as demonstrated above by calculation. Each system, which varies (as the figures show) with

the constructor, who has generally followed the advice of the chemical engineers of the industry in regard to such and such an improvement, will first be studied.

Type I.—Triple effect of 150 square metres of heating surface (this example is taken as representing the average build of this type) combined simply with an injection condenser and a pump or wet-air vacuum. Evaporative capacity 3,000 litres (660 gallons) per hour, say 20 litres (4·4 gallons) per hour per square metre. In this case it is plain that the steam economy rests in the number of vessels. The condensed water from Nos. 2 and 3 going direct to the condenser mix with the injection water. Consequently it is totally ejected by the condensed

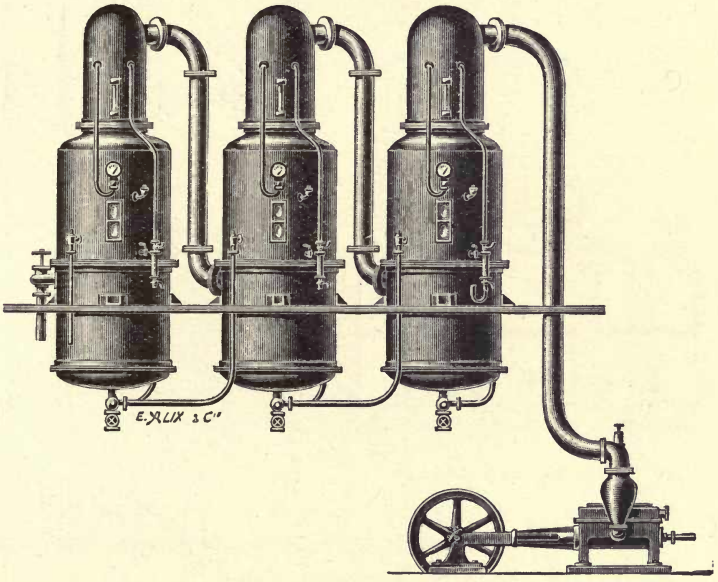

FIG. 95.—Triple effect evaporation plant.

water pipe. This arrangement (Fig. 95), which is the most simple, ought not to be used unless unavoidable; it should be completed by a condensed water pump as will be shown farther on (Type III.).

Type II.—Triple effect of 150 square metres (three equal vessels of 50 square metres) combined (1) with a countercurrent barometric condenser; (2) with a condensed water pump; (3) with a dry air pump, tandem system by direct motor. The steams from Nos. 1 and 2, condensed in the heating chambers of Nos. 2 and 3, are aspirated and collected by a special condensed water pump, which maintains the vacuum in the ebullition or evaporation calandria of Nos. 1 and 2, by means of

suction valves. This condensed water pump, 150 mm. by 320 mm., with four valves, two for suction and two for propulsion, all made of bronze, do not propel further than a metre, so a *capot*, about the level of the propulsion pipes, receives the condensed water from Nos. 2 and 3 vessels, to be afterwards propelled by a special (bronze) centrifugal pump to the feed condenser, to be utilised in the maceration of the wood. The

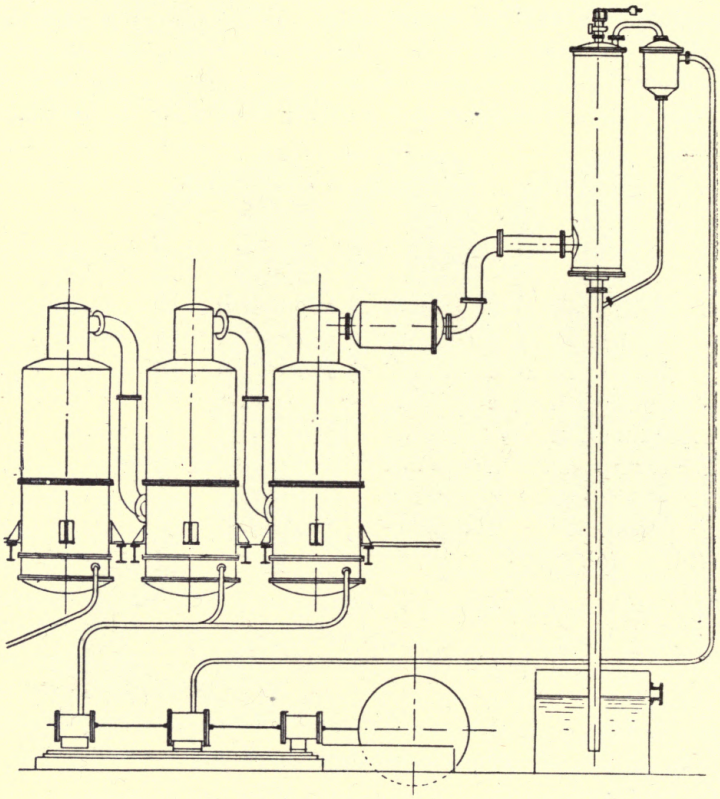

FIG. 96.—"Triple effect" connected with its barometric condenser, its dry air pump and condensed water pump in tandem (Bonnet-Spazin).

copper condenser is of a new type, countercurrent with perforated baffles 650 mm. × 3 m., water separator and injection valve. It is completed by a barometric column 250 mm. in diameter and 12 metres in length. The air pump with direct horizontal motor, bayonet system, 250 mm. × 320 mm., air cylinder 300 mm. × 320 mm. Fig. 96 represents the plant on the scale of 1 : 50, such as it works in certain factories where it yields good results. Although the fixing of the baro-

metric condenser requires a minimum height of 11 metres it may be said that it works normally in all its applications.

Type III.—Triple effect of 150 metres combined with (1) a wet air pump and injection condenser, which works, in parallel, a condensed water (from Nos. 2 and 3) pump; (2) an automatic feed appliance (Koerting's) placed to the left of Fig. 97, propelling directly to the steam boiler the blow-off water from No. 1. Fig. 97 shows the arrangement which is, in fact, the average modern one. As will be seen, each vessel

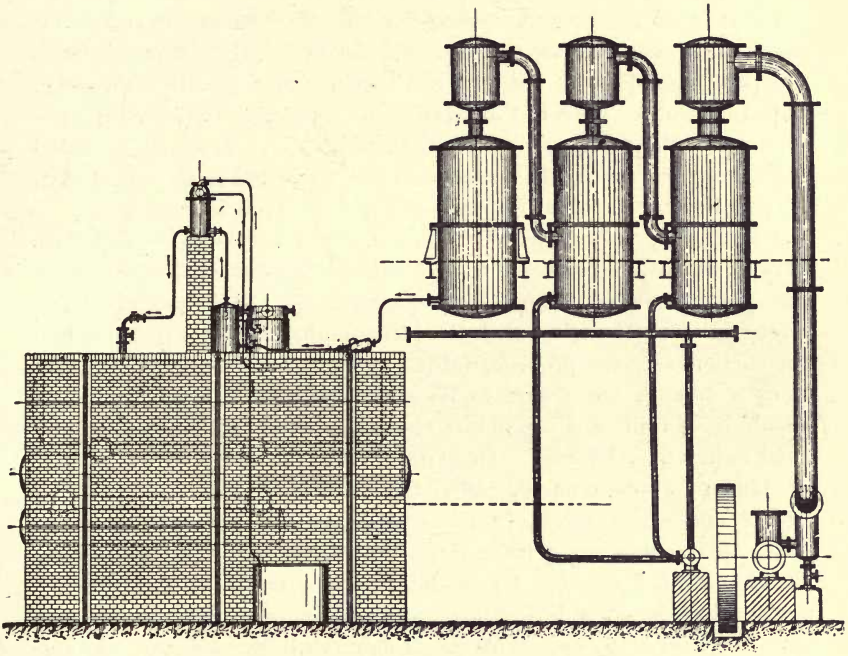

Fig. 97.—"Triple effect" combined with moist air and condensed water pumps, the water blown off from No. 1 being propelled to the boilers by Koerting's automatic feed.

is fitted with a Heckmann's safety vessel, which suppresses the old arrangement, the utility of which has been justly disputed. To complete this plant, it is necessary to add a juice reheater such as shown in Fig. 90, the economy in steam realised by which has already been figured.

Type IV.—Finally, and it is this which the authors would like to see adopted more frequently instead and in place of the injection or barometrical condenser, a surface condenser, capable of condensing all the tannic vapours. For it must not be forgotten, that the vital question in tannin manufacture is the purity of the maceration water,

and besides, the calories recuperated correspond to an appreciable economy in steam, which results in regular, constant, and, so to speak, automatic working. Thus, in adopting surface condensation, Derosne or tubular type, in which the tannic vapours from No. 3 circulate and completely condense, so as to allow the incondensable gases only to pass to the dry air suction pump, a notable improvement will be realised in present plant. Practical data enable the authors to assert that, in the case of a triple effect of 150 square metres, a tubular condenser of 65 square metres, in which there circulates as refrigerating agent not the juice as in the Derosne system, but the water coming in abundance from the liquor coolers, will suffice. Moreover, this arrangement is only complementary to the juice reheater placed originally at the steam exit from No. 3. This last installation is completed by the addition of a pump for the condensed waters from Nos. 2 and 3, which, collected with those from the surface condenser, are propelled to the maceration passing through the feed reservoir of the vats or autoclaves.

Air Pumps. Condensed Water Pumps. Wet Air Pumps.—Types are very numerous, and it would take too long to enumerate them here, but pumps offering the greatest guarantee in simplicity and in the strength of their organs, with the least number of valves possible, two for suction, two for propulsion, their speed not exceeding fifty revolutions a minute, are always to be recommended. Finally, the pump should have a direct steam motor, the escape steam from which can be used to heat No. 1 vessel. This same pump will work, either parallel or tandem, another pump, called the condensed water pump, all of bronze, and which serves, in the case of a double effect, to draw the condensed tannic water from No. 2, and in the case of a triple effect, that from No. 2 and No. 3 vessels. Figs. 90, 93, 96, 97 have already shown the position and the shape of these pumps. Two pumps are given here: Fig. 98 represents an air pump with direct motor, and Fig. 99 a vertical twin made by Robatel, Buffaud & Co. of Lyons.

Dry Air Pump.—This type of pump, which is used in working with a barometric or surface condenser, is generally coupled tandem with the preceding. It also is always driven by direct motor; its air cylinder has drawer distribution and arrangement for the suppression of injurious spaces. There is an advantage in constructing this pump altogether in bronze, in the same way as the condensed water pumps; as to the wet air pump, the interior organs—rod and piston, cylinder jacket, valve seats—should or can be all in bronze.

Triple Effect Plant. Starting and Working of Type III. Testing Tightness.—It is necessary about every fortnight, and every time that a defect is noticed in the working of the plant, to make a tightness test,

to make sure that no air enters and that all the joints are tight. The test is carried out as follows. Close all the taps which place the vessels in communication with the open air: the large steam escape taps, the live steam taps, the discharge taps of each vessel, the "butter" tap.[1] Make full joints on the blow-off pipes of No. 2 and No. 3 and on the collar which connects with the double condensed water pump. Close the water inlet tap to the condenser, fill the cylinder and the upper evacuation chamber of the air pump with water. Open the three taps for blowing off incondensable gases. Close the liquor inlet taps of No. 1 and leave the others open; that done, start the air pump to work, so as to produce a vacuum in the three vessels; increase this vacuum as much as possible. If the piston is in good working order, as well as the glands of the stuffing boxes, 65 to 67 cm. of vacuum may be easily obtained.

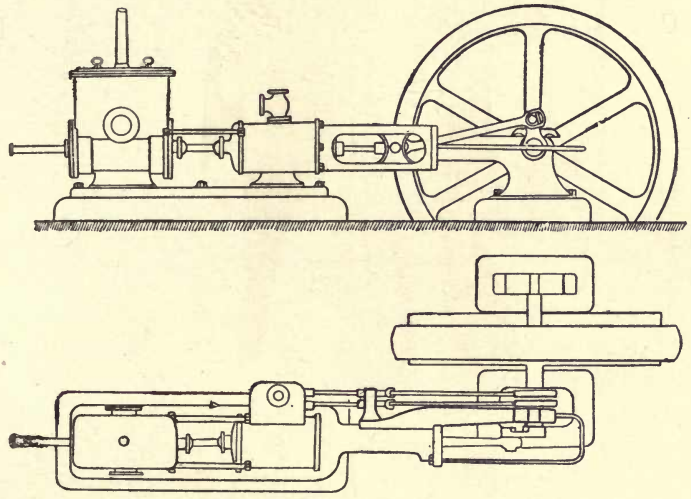

FIG. 98.—Moist air pump with its steam motor.

The pump is then stopped. If the vacuum falls very slowly, it is a proof that all is tight; if the vacuum falls quickly, it is a sign of a leak in one of the vessels or in the piping. It is necessary to find the leakage; the whistling which occurs when air enters anywhere is a convenient indicator to find it. One can, moreover, ascertain from which vessel the leaks take place, by closing first the liquor tap and that for the evacuation of the incondensable gases from No. 2, thereby isolating No. 1. If the fall of the vacuum stops in No. 1 vessel, that is a proof that the leak does not occur in that vessel; if, on the other

[1] The *robinet à beurre* supplies oil to float on the surface of the liquid, being evaporated so as to act as a froth preventive.—D. G.

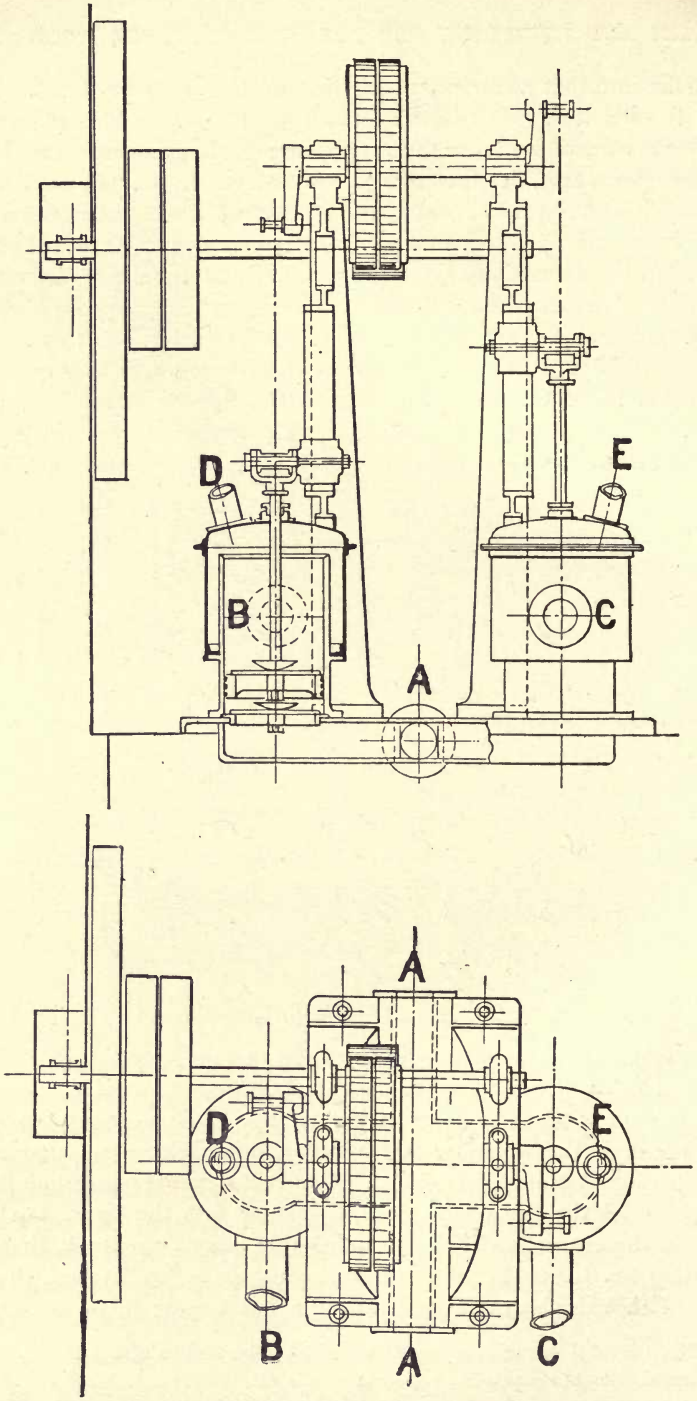

FIG. 99.—Twin wet air belt driven pump. AA, aspiration pipes for water and gases; BB, discharge pipes for condensed water; DE, incondensable gas pipes.

hand, it increases, then the leak is to be found in that vessel. No. 2 vessel is isolated in the same way, closing the liquor tap and the tap for the evacuation of the incondensable gases of No. 3. It must be well realised that any entrance of air leads to the bad working of the triple effect, and that it is, therefore, necessary to correct it at once.

Starting.—When tightness is secured a start is made as follows. Care is taken to remake the joints on the blow-off piping of No. 2 and No. 3 on the double condensed water pump. Close the liquor inlet taps of the three vessels; open, to its normal point, the water inlet tap to the condenser. Start the air pump; a vacuum is made in the three vessels, and when it reaches 30 cm. in No. 1, and 64 in No. 3, open the liquor feed tap in that vessel. Let the juice run in to the level of the tubular plate. Close the feed valve. At the same time turn slightly the escape valve of the engines, so as to turn the escaped steam on to No. 1; the liquor in No. 1 soon commences to boil. Then open the feed valve of No. 2 to admit the liquor into that vessel. At the same time, again, open the tap of No. 1, to maintain its level. Let the liquor run into No. 2, until the boiling froth appears at the glass, and then open, finally, the feed tap of No. 3, to admit the liquor. As in No. 2, the liquor is allowed only to reach the top of the tubular plate. During the whole time of this period of feeding continue to heat No. 1, so as to maintain therein the vacuum at 35 or 40 cm.; to do that it is necessary to increase gradually the opening of the escape valve of the engines, or to apply direct steam. Continue likewise to increase the quantity of liquor in No. 1 and in No. 2, but taking care not to allow the level of the liquid to rise too high. Starting from the moment when No. 3 is fed, the ordinary working of the triple effect commences. The following rules must then be observed:—

Rules to Follow in Ordinary Working of Triple Effect.—The two essential precautions in the working of the triple effect lie in the heating of No. 1 and in maintaining the level of the liquid in the three vessels. No. 1 must be heated, more or less, so that the vacuum is maintained uniformly at 35 or 40 cm. In the other vessels, the vacuum establishes itself, and working is normal when there is a vacuum of 50 to 55 cm. in No. 2 and 65 cm. in No. 3. The levels must be kept at such a height that there is no entrainment in any of the vessels, *i.e.*, so that the froth does not rise too high and so be entrained into the safety vessel. Daylight must always be seen between the front peep-glass of the vessel and that above the bottom behind. If boiling be too violent in one of the vessels, the feed should be reduced, so as to lower the level. If the liquor be at any moment seen to prime, which may be seen by aid of the peep-glass on the safety

vessel, the air tap is opened a little so as to lower the vacuum a little in that vessel, the effect of which is to stop the boiling at once. At the same time the feed of that vessel is reduced. Care must be taken in reducing the feed of a vessel to reduce that of those which precede it in the same ratio, otherwise the level of the latter will ascend. The vacuum in No. 3 should be 65 cm. That point is got by appropriately adjusting the water inlet tap of the condenser. It is evident that the amount of water injected should be capable of condensing the steam from No. 3. The temperature of the mixture of hot water, which is ejected by the pump, ought to be 30° to 35° C. at the maximum. It is evident that in winter less water for condensation will be necessary than in summer. If, in spite of regulating appropriately the volume of water injected, the vacuum does not stand at 65 cm., the condition of the segments of the piston of the air pump of the stuffing boxes with glands and the rubber valves must be ascertained or it may be necessary to make a tightness test. Care must be taken to see from time to time that the blow-off cocks of each vessel are in good working order. The concentration of the liquor is greater in proportion as the evaporation progresses in No. 3.

Sampling.—Samples are taken by aid of a special appliance adapted to each water-level as follows. Close the lower and open the upper tap of the reservoirs. When the reservoir is filled with liquor, close the upper tap, which brings the interior of the reservoir in communication with the atmosphere by the small air-hole; the juice flows into the sample vessel, in which it is tested by dipping a hydrometer into it. To aspirate that juice, open the two taps and close the top one, when air bells are seen to rise up the water-level.

Discharging the Extract.—When the liquor in No. 3 is of the desired strength, it must be extracted, which is done by aid of the juice pump or the "syrup discharger" (*vide-sirop*). Close the three liquor inlet taps on the three vessels and turn off the steam heating No. 1, allowing the vacuum to fall in No. 3 to aid the discharge. The piston rod of the extract pump is locked, and the discharge tap of No. 3 is opened or the syrup discharger is brought into gear. When that vessel is empty the discharge valve is closed, the pump unlocked and No. 3 vessel is fed through No. 2, and No. 2 through No. 1, thus resuming normal working. Care must be taken that the bulk of the mass boiled in No. 3 does not rise 10 cm. (say 4 inches) above the tubular plate, so as to avoid loss of juice by the condensed water pumps and the dial tap.

Stopping the Triple Effect Plant.—In stopping, the feed taps of each vessel must first be closed. The steam heating No. 1 must be

turned off by turning the escape valve so as to blow off in the open air, and the air pump is stopped.

Restarting.—Set the air pump to work. Open the steam inlet tap so as to turn heating steam on to No. 1, and when the boiling has resumed its usual course the juice feed is turned on.

Cleaning.—Care must be taken, so as to maintain the same output always, to clean the tubular bundle and wash the vessels at least once a month. It must be well realised that the encrustations on the inside of the tubes considerably diminish the turnout of the apparatus, and that it is very desirable to remove these encrustations, as often as practicable, to prevent them from hardening too much. To clean No. 1 brushing and washing with hot water often suffices, likewise for No. 2. In No. 3 the encrustations become very hard, and it is then necessary to use a special apparatus, called the turbine brush. To facilitate the work of cleaning, water may be boiled in the vessels; to do that it suffices to fill them with water up to the level of the plates and to turn on live steam into the heating chambers. This softens the hard encrustations and aids their removal by brush and scraper. The wash waters are evacuated through the wash collector, which discharges them down the drain.

Kestner's Evaporator.—The evaporation plant in common use has now been reviewed. A new system, based on a principle but little known, by which a much greater output and an absolutely rational working of the plant is realised, now falls to be described. These evaporators are comparatively recent, but their applications multiply from day to day in all industries where evaporation has to be effected. The evaporators referred to are those of Kestner of Lille. This plant is constructed so as to be heated by live steam from the boilers or escape steam from the engines; they are simple, double, triple or multiple effect; concentration is effected in tubular bundles 7 metres in length. The liquor, distributed in the lower part of the plant by a special arrangement, divides itself equally into all the tubes of the bundle, conditions which realise a great advantage from the point of view of damage to products sensitive to heat; as is the case with wood extracts, from the point of view of the liquid in circulation, the differences are remarkable compared with other systems; thus for two sets of plant doing the same work an ordinary quadruple effect of the type previously described contains 15,000 litres in concentration, whilst Kestner's plant only contains 500! This difference clearly indicates that a new principle is applied in this evaporator; it is the phenomenon termed "grimpage" or "climbing" which causes the liquid to rise in tubes 7 metres long. If the ratio be established between the volume of the liquid which

passes up the pipe from bottom to top and the volume of steam which escapes from the top of the tube, it is found that this proportion is at least as 1 to 1,000. Globules are formed in the bottom of the tube, but when the volume and the speed of the steam are sufficient the liquid assumes an ascending motion, in the form of an extremely thin layer along the interior side of the tube, whilst the steam circulates with a considerable speed of 25 metres per second in the axis of the tube (see Fig. 100). It will be realised that in these conditions evaporation is highly intensive and that the liquids are actually placed in the best conditions to prevent them getting damaged. Practically in all cases where Kestner's evaporators have been applied more superior products have been obtained, and the reason why the authors recommend this particularly interesting plant is because they have found such to be the case.

Description of Fig. 100.—A multiple-effect vessel, on Kestner's plan, consists of a copper tubular bundle enclosed in a steam chamber or calandria; in the lower part of the calandria, of a distributing case of the liquid to be concentrated, which is fed into it by a single pipe of small diameter; in the upper part of the steam separator. The tubular bundle, R, is 5 to 7 metres long, and the tubular plates on which the tubes are mandrilled are of bronze. The body, M, of the calandria is of wrought iron or copper. The separator, S, is spherical and comparatively small. It contains a baffle, D, fitted with blades, similar to those of a centrifugal turbine. This baffle is fixed; the blades impart to the steam, which must pass between them, a rotary motion, which separates the liquid drops entrained by the steam. Fig. 100 shows a Kestner evaporator working at atmospheric pressure. The liquid separated from the steam flows away through L into an upper tank, Y. If the plant work *in vacuo,* the pipe, L, is prolonged into the lower part of the vessel, below, for example, the floor supporting the weak liquor reservoir, X, and the flow of concentrated liquid occurs barometrically in the vessel, Y, resting, in that case, on the ground. In vacuum working, the steam exit pipe, B, from the concentration, is connected with a safety vessel, communicating with a barometric condenser, or with a wet air pump if certain factory arrangements hinder barometric condensation, which is the best from all points of view. The heating steam enters through A. The condensed water exit is in the lower part of the calandria. The steam and the liquors to be concentrated circulate in opposite directions. Independently of the above advantages, Kestner's plant possesses, moreover, general simplicity in construction; few joints, consequently few leaks; and slight entrance of air in plant working *in vacuo;* also easy regula-

tion of the plant; the delivery of the feed pipe of No. 1 is regulated, according to the density to be obtained in the last vessel. The floor space required is small.

Washing.—Washing is a peculiar feature of this plant. It enables it to maintain its maximum output. The section of a vessel (Fig. 100)

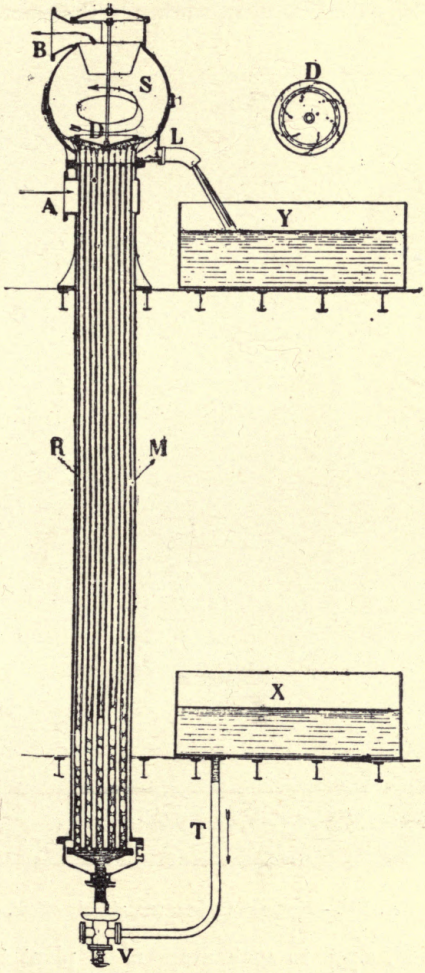

FIG. 100.—Section of a Kestner evaporator.

shows that, as soon as the plant stops, the liquid may be entirely evacuated. Each vessel may be washed very rapidly with the hot condensation water; such washing requires about a quarter of an hour, and entails no stoppage of the factory. The wash water circulates in the same di-

rection as the concentration liquor; cleaning is, therefore, particularly efficacious, and the plant, it may again be repeated, works with a maximum yield, whilst, in all other evaporation systems, cleaning of the plant is a lengthy operation that cannot be done daily, which thus increases the difficulty. One or more Kestner vessels may be added to existing vessels. Their heating surface is thus increased and their

Fig. 101.—Kestner evaporator, simple effect.

output augmented, since to such evaporation plant there have been added one or several elements of much superior evaporative capacity. Even in heating by a steam coil, a Kestner evaporator, fed in the first instance by weak liquors, would supply steam to the coils to finish the concentration, and would thus realise very great economy of steam by establishing a group, working by double effect, the first element of which would be of great working capacity. Kestner's plant has

numerous applications in different industries connected with concentration, and particularly as regards wood extracts, tanning extracts, logwood extracts, etc. Figs. 101 and 102 are photographs of plant actually at work.

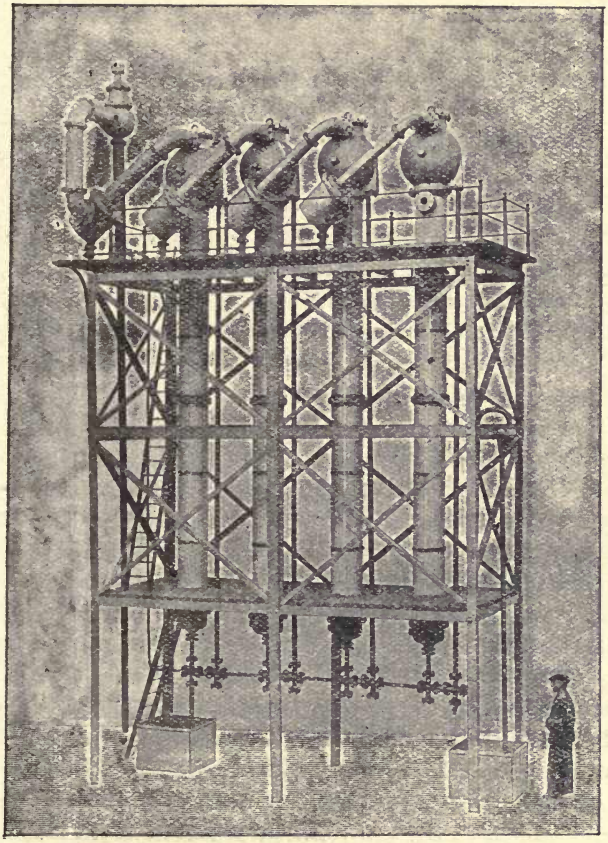

FIG. 102.—Kestner evaporator, quadruple effect.

Summing up, according to what has just been said, Kestner's new evaporator is certainly the apparatus best adapted, from all points of view, for the manufactures with which we are concerned, and that is why it has been described at greater length and recommended by the authors. Moreover, its efficiency has been proved.

CHAPTER III

MODEL TYPE OF AN EXTRACT FACTORY. CAPITAL REQUIRED. CALCULATION OF COST PRICE

Specification of a Model Factory.—As a type of an extract factory, the authors take one in which the plant and equipment enable 60 tons of chestnut or oak wood to be treated in twenty-four hours, that is, in fact, an ideal industrial remunerative factory. The following is a descriptive list of the various items of plant required for this manufacture on such a basis; it includes:—

I. *Autoclaves.*—Five autoclaves of 10,000 litres (2,200 gallons) capacity, 1·85 metres in diameter and 4 metres in height, each including one charging manhole, 400 mm.; one discharging manhole, 800 mm.; one steam inlet tap, 70 mm.; one water feed tap, 90 mm.; one escape steam tap, 80 mm.; one three-way valve of 100 mm. for transfers; one water-level; one pressure gauge; one bronze case with valve and snifting valve; packing for all the orifices; piping connecting all the autoclaves to each other; cast-iron spout for feeding the chips, with hinged lid; chain with pulley and counterpoise for working the top lids; woodwork and columns staging to support the autoclaves. Such autoclaves are truncated in shape as in Fig. 83.

Ia. *Battery of Wooden Vats.*—The above battery of autoclaves is given as a matter of form only, as it does not admit of the rational working (in the case of factories which wish to make other extracts than chestnut) recommended by the authors, from knowledge of the facts, which therefore requires a battery of sixteen wooden vats of 20,000 litres (4,400 gallons) capacity, capable of holding 5 metric tons of chestnut or oak chips and represented by Fig. 80. This battery when fully installed is a little cheaper than an autoclave battery, say . . £2,600

II. *Cutters.*—Two cutters with angular *tourteau* (boss), each capable of reducing 30 metric tons of wood into chips in twelve hours, comprising: Oak foundation, steel shaft, three lubricating bearings, cast-iron boss bearing the blades,

Carry forward £2,600

Brought forward £2,600

fast and loose pulleys, fly-wheel, chute or spout, spur, wrought-iron hood, two sets of knives, £140 each, say the pair 280

III. *Elevating Conveyer.*—Two elevators; one conveyer 200

IV. *Steam Engine.*—One non-condensing high-speed steam engine, horizontal type, 50 to 70 h.-p., to work the two cutters, the elevator-carriers and the various shaftings. Erection 480

V. *Boilers.*—Two multitubular steam boilers, horizontal type, of 120 square metres of heating surface, weighted to 8 kilogrammes, fitted with their accessories: door fittings, sweeping brush frames, boiler supports, dampers and their working appliances, safety valves, pressure gauge, water-level, feed tap and valve and appliances for being heated by a gas generating furnace, burning exhausted chips, £600 each, say the pair 1,200

Two gas generating furnaces complete, each £120, say the pair 240

VI. *Triple Effect.*—One triple effect evaporation plant, with a heating surface of 180 square metres capable of evaporating 3,500 litres (770 gallons) per hour, consisting of three copper vessels, tubular bronze plates, Heckman's safety vessel and steam communication pipe. With complete piping, *viz.*, valves for escape steam and live steam, for charging and discharging liquor level, sampling vessel, "butter" tap, peep-glasses, manhole, condensed water discharge pipe and froth return pipe. One copper condenser on the air pump. One air pump with direct motor, coupled to a pump for the evacuation of the condensed water from Nos. 2 and 3 vessels, enabling this water to be used for feeding the autoclaves. One liquor pump for emptying No. 3. One wrought-iron collecting globe of the escape steam from the various engines, with inlet and exit pipes, blow-off cock and safety valve. All complete, with the piping connecting the three vessels with each other and that connecting the latter with their respective pumps, as well as all the woodwork and masonry expenses, in all . 3,200

VII. *Mechanical Decantation of the Juice.*—Four decanting turbines of 1·2 metres in diameter, with an output of 1,000 litres (220 gallons) per hour, all complete in copper

Carry forward £8,200

LIBRARY OF THE

Brought forward	£8,200
or bronze as regards the parts in contact with the juice, Robatel, Buffaud & Co.'s system; each £160, say for the four	640
VIII. *Factory Buildings.*—One hall 700 square metres in area, with saw indented metallic framework and slant roof for the chip-granary, plus a shed of 400 square metres for the extract vats, cooper's shop, the office, laboratory and a small repair shop, the whole built of material to be found on the spot at the inclusive price of	1,600
IX. *Chimney with Mechanical Draught.*—One chimney with Sturtevant converter	200
X. *Site.*—Approximate price of site, which ought moreover to be selected on a spot fulfilling the conditions required for this class of work: contiguous to a junction, from which a network of branch lines emerge with a water supply of at least 50,000 litres (11,000 gallons) per hour, the geometrical centre, as far as possible, of chestnut or oak forests, the area of which enables the factory to be written off as depreciation in ten years or sooner if the amount of depreciation is a factor of the cost of buying wood at a price not exceeding twelve shillings a ton, finally minimum and active hand labour.	
The authors estimate a total area of 4,000 square metres as sufficient for such an installation at three francs the yard, say for purchase of the site	480
XI. Water works and its pump, trucks and Décauville track, accessories and sundry articles for the manufacture .	320
XII. Five tubular refrigerators	320
XIII. Vats for liquor, etc.	160
XIV. Office and laboratory	80
XV. Noyer's condenser-reheater reservoir . . .	160
XVI. Shafting and different pulleys	80
XVII. Wood in shed	800
XVIII. Incidentals for casks, weighing machines, equipment, etc.	160
Say, for starting a factory treating 60 tons of wood in twenty-four hours, a capital of	£13,200
Add for working capital	800
which then represents the total maximum capital for starting a chestnut extract factory on the above-mentioned basis, that is to say capable of producing a minimum of 13 metric tons of extract in twenty-four hours	£14,000

Cost Price.—For a capital of £14,000 and on the basis of treatment and production indicated above, the cost price of 100 kilogrammes of extract 25° may be dissected thus:—

(1) *Raw Materials.*—500 kilogrammes, say half a ton of chestnut wood (45 per cent. water), at twelve shillings the ton on spot	£0 6 0
(2) *Labour.*—Gratuities, labour, accident insurance	0 1 4·8
(3) *General Expenses.*—Rent, rates, taxes, insurance, interest, depreciation	0 1 2·4
(4) *Working Expenses.*—Repairs, electric light, clarification	0 1 0·0
Say per 100 kilogrammes (2 cwt.) of unpacked extract at the factory	£0 9 7·2
Taking the high price of casks into account, allow for packing 100 kilogrammes (2 cwt.) of 25° extract	0 2 4·8
The average cost (free) on rail of freight into France or European port	0 2 9·6
Commission to sellers	0 0 4·8
Say per 100 kilogrammes (2 cwt.) of 25° extract delivered free to customers	£0 15 2·4
Now the average selling price, which will remain stationary for a long time, with a tendency to a rise, is	0 17 7·2
Say a net profit per 100 kilogrammes (2 cwt.) of 25° extract	£0 2 4·8

Hence taking the working year of 300 days, with a 60 ton factory as type and erected with a capital of £14,000, will give a return of $\frac{2\text{s. } 4{\cdot}8\text{d.} \times 13{,}000 \times 300}{100}$ = £3,978 16s., or a gross profit of 30 to 33 per cent. [But on the authors' own showing the yield from 500 kilogrammes of wood is only 100 kilogrammes of extract, that is one-fifth, and the fifth part of 60 metric tons or 60,000 kilogrammes is 12,000 kilogrammes, not 13,000.—Tr.]

Generally it is necessary to take into account the increase in the prime cost of wood, but in any case it must be taken that the redemption is effected in three or four years; the cost of the ton of wood generally rises from 15 to 17 francs (12s. to 13s. 7·2d.) or more, the profit per 100 kilogrammes of extract then falls from 30 to 33 per cent. to 10 to 12 per cent., a net profit still appreciable in these times, because, in fact, it must not be lost sight of that the tannin industry, although of French origin, is perhaps at the present day one of the most pros-

perous, and it may be said that it will be so for a long time if it gives greater and greater satisfaction to its customers, the tanneries, which are following a progressive march in virtue of present eventualities, which will create for a long time to come new needs which will perforce react on the European market.

CHAPTER IV

STATISTICAL

Number and Capacity of Extract Factories in France, Corsica, Italy, Spain —Total French Production in 1904*—Imports and Exports of Chestnut Extract and of other Vegetable Liquors into and out of France since* 1900*, according to Official Documents*

Number and Capacity of Extract Factories in France.—In 1875 there were only seven factories of this kind, to-day there are more than twenty. They are tabulated below.

Name of Manufacturers.	Locality of Works.	Tons of Wood treated per 24 hours.
1. Liebaut & Co. .	Ossez (Basses-Pyrenees) . .	60 tons chestnut
„ „	Nay („ „) . .	50 „ „
„ „	Tournaye (Htes.-Pyrenees) .	100 „ „
„ „	St. Nicholas (Morbihan) . .	50 „ „
2. J. P. Rey . .	La Rochette (Savoie) . . .	50 „ „
„ „	Couze (Dordogne)	120 „ „
3. Levinstein Co. .	Cornil (Corrèze)	100 „ „
4. Watrigant fils .	Pont Vert (Cher)	100 tons chestnut and oak
„ „	Marquette-lez-Lille	50 tons quebracho, sumac, myrobolam
„ „	„ „ „	30 tons logwood
5. Rey frères . .	Montreuil-s.-Ille (I.-et-V.) .	90 „
„ „	„ „ „ . .	30 „ oak
6. Philippe & Co. .	Lalevade d'Ardèche	120 „
7. Huillard & Co. .	St. Denis-des-Mures (Hte.-Vienne)	60 „ „
„ „	Suresne (Paris)	sumac, quebracho, logwood
8. Marchal - Courbaize . . .	Maurs (Cantal)	60 tons
9. Gillet et fils .	Lyon-Vaise	30 „
„ „	Molières-Cavaillac (Gard) . .	30 „
10. E. Roy & Co. .	Lanouaille (Dordogne) . . .	50 „

Name of Manufacturers.	Locality of Works.	Tons of Wood treated per 24 hours.
11. Miallon & Co. .	Banassac (Lozère)	50 tons
12. Société des Tannins concentrés	Montrejeau (Hte.-Garonne) .	50 „
13. Roubin & Co. .	Lalevade (Ardèche)	30 „
14. Ausset & Hermet	Genolhac (Gard)	30 „
15. Société des Produits chimiques . . .	Labrugnière (Tarn)	20 „
16. Abeille . . .		20 „
17. Croniere-Querelle . . .	Ponteils (Gard)	20 „
18. Bordet & Co. .	Froidvent (Côte-d'Or) . . .	20 „ oak
19. Luc et fils . .	Nancy	20 „ „
20. Baux	Marseilles	10 „ chestnut
21. Patre et Mabillat	Factory transferred into Italy.	
22. J. Noyer & Co.	Factory projected at Lavaulte-sur-Rhone.	

Say a total, therefore, of twenty chestnut extract manufacturers and twenty-six factories of chestnut extract, of which three manufacture especially oak extract. The total consumption of this aggregate of factories, and, therefore, their vitality, has been given in the article, "Deafforestation of Chestnut Woods in France". The authors, however, believe that, in spite of their calculations based on official figures, the life of these factories will be much shorter, because the factor, which might be called reserve, is ignored or is capable of too much approximation for it to be possible to be affirmative thereanent.

Italian Factories.—Lepetit-Dollfus et Gansser at Garessio (Cunes); Martinolo et Lamberti at Millesimo (Savona); Giuseppe Massa at Garessio; Varaud et Paret at Bagni di Lucca (the two last factories work for exportation *viâ* Hamburg); Patre et Mabillat at Oneglia; Società Anonima Estratti Tannici Darfo at Milan; Fratelli Dufour at Geneva; Società Italiana di Estratti conciandi e colorandi at Villafranca Lun, near La Spezia.

Spanish Factories.—Torrabadella fils at San Martin de Provensals, near Barcelona; Brillas, Pagans y Ca. at Cebra, near Gerona.

Corsican Tannin Factories.—From a visit to Corsica in July, 1904, made at the instance of an important factory of that country, the authors brought back certain particulars as to the situation and size of the

tannin factories of that department, from which, moreover, they drew conclusions which fully confirm those of M. Donati on the deafforestation of Corsica. The following are some data as to the four existing factories :—

I. The factory of L. J. Levinstein & Sons, of Berlin, is situated near the station of Casamozza (commune of Lucciana) and on the banks of the Golo, 22 kilometres from Bastia ; built in 1902 it produces in twenty-four hours about 20 tons of 25° extract.

II. The factory De la Société Corse, for the treatment of wood, is situated near to the Golo and the station of Barchetta (commune of Volpajola), at 30 kilometres from Bastia. This factory produces 18 tons of 25° extract in twenty-four hours. It is only 8 kilometres distant from the Casamozza factory.

III. La Société Anonyme de Champlan includes the Champlin factory itself (the old Serrières de Lyon-Vaise factory), 8 kilometres from Folleli-Orezza, where the same company has just installed a second factory, which is 32 kilometres from Bastia. The joint production of these two factories is 20 tons of 25° extract per twenty-four hours.

From the foregoing it will be seen that all the factories are near to one another, within a radius of less than 20 kilometres, say 12½ miles. Their means of working, as far as a supply of wood is concerned, are therefore perforce paralysed, and the competition engendered thereby causes the price of chestnut wood to be raised, so that it reaches 9½ francs the stère of 460 kilogrammes, say at the most 20 francs (16s.) the ton. These factories are all on the east coast of Corsica, a district where the chestnut grows, but also one which is dominated by malaria, especially in the low-lying marshy parts, which every now and then overcomes the working staff of these factories, which thus experience, by that fact alone, a certain difficulty in recruiting the labour, which otherwise leaves much to be desired as regards the work done. This dangerous zone is, however, planted with eucalyptus, especially on the departmental railways passing through the above districts. From the total quantity of extract produced by these four factories it will be seen that the daily consumption of chestnut wood exceeds 350 tons, that is to say 210 tons more than in 1901, the year in which Donati announced that the deafforestation of Corscian chestnut trees would be complete in fifty years : according to the authors' figures, it will be seen that the complete devastation of the chestnut forests will be accomplished in a much shorter period of time, and which may be fixed within a few years only.[1] The whole production of these factories is

[1] The authors learn that Corsican deafforestation will soon force certain tannin factories to engage in the sugar industry, and already beet-root experiments have been organised at Casamozza, near Bastia.

shipped from the port of Bastia, the Corsican consumption being *nil* as it possesses no tannery.

Total French Production, 1904.—The total production of extract including that of Corsica reached in 1904 about 105,000 tons, representing a value of about 26,000,000 francs (say £1,040,000).

TABLE XXIV.—TANNIC ACID EXTRACT TRADE OF FRANCE, 1900-1903 (BOTH INCLUSIVE)

Country from or to.	Imports from. Net Weight in Kilos.[1]				Exports to. Net Weight in Kilos.[1]			
	1900.	1901.	1902.	1903.	1900.	1901.	1902.	1903.
Britain	69,974	57,223	38,095	...	12,976,890	12,210,233	12,681,424	19,045,474
Holland	61,537	93,026	...	...	...	...	...	...
Germany	...	85,278	150,850	116,568	3,182,555	4,821,441	4,516,444	6,688,161
Belgium	381,460	137,391	321,315	196,526	6,171,819	5,629,341	6,438,218	7,963,784
Switzerland	118,842	108,418	122,436	118,273	680,214	557,036	648,289	637,331
Austria-Hungary	493,696	883,822	707,750	666,383	...	736,216	...	...
Spain	...	...	...	...	314,513	...	...	718,961
Italy	137,400	183,285	51,201	48,867	773,192	872,596	1,068,239	1,303,514
Roumania	...	...	...	...	...	...	667,572	...
Russia	...	...	...	...	476,527	1,347,269	1,986,038	1,057,057
U.S.A.	111,030	86,214	137,903	115,267	...	...	...	...
Argentina	...	...	30,082	42,931	...	...	...	...
Uruguay	...	...	...	32,315	...	...	...	...
Other countries	108,832	5,262	...	53,887	1,412,554	1,939,014	2,007,615	1,978,680
Algeria	...	...	...	...	24,782	37,744	38,265	11,240
Tunis	...	...	...	...	...	...	20,624	42,800
Martinique	...	...	...	...	27,619	...	...	...
Réunion	...	...	...	...	...	31,489	...	...
Other colonies	...	...	...	...	26,699	27,785	20,099	13,988
Yearly total	1,484,771	1,639,919	1,587,457	1,416,745	26,066,364	28,210,164	30,092,727	38,460,990
Yearly value(francs)	297,954	327,984	317,491	283,349	5,213,273	5,642,031	6,018,545	7,892,198

Metric Quintals (100 kilos.). Value.

Year 1904, Importation 19,625 334,000
" Exportation 522,740 8,887,000

The "1904 results as far as known are given as gross weights.

[1] By converting the numeration sign the furthest to the right into a decimal sign the weights are then expressed in metric tons of 2,200 lb. and decimals thereof.

From the preceding figures and since 1900 it is easily seen that the (French) imports have only increased 37,000 francs, say 9,000 francs per annum, whilst the exports have increased by 3,673,000 francs (£146,920) or by 900,000 francs (£36,000) per annum, an increase which justifies moreover the impulse given to this eminently French industry.

CHAPTER V

METHOD OF USING CHESTNUT EXTRACTS IN TANNING

THE tanner ought always, unless he makes them himself, to strive to secure the best quality of the chestnut extracts offered to him, which should always contain at 25° B. a minimum of 28 per cent. of tanning substances assimilable by the leather (analysed by the official method of the International Association of Leather Chemists), be soluble in water without more than $\frac{1}{2}$ per cent. of insoluble and free from all trace of chemicals or other body foreign to tannin. The purified brands, clarified and rendered soluble by absolutely mechanical processes, that is to say those in which all the insoluble extractive matter is eliminated (which, in ordinary extracts, is precipitated as soon as water is added and forms a mud suspended in the solution), should be preferred as giving the maximum yield in leather. This kind of extract, soluble in the cold state, is used for tanning heavy leather in the pit or large ox-hides for belting ; precise instructions will therefore be given as to its use in this special department of tanning farther on. The purified extracts, clarified by filtration of the liquor, according to the authors' instructions, and with addition of borax, are especially adapted for semi-rapid or accelerated tanning and are even used for tanning cow-hide, calf, sheep and goat skins, into which they enter as adjuncts to, rather than as substitutes for, oak bark. The advantages of this method are esteemed for economical reasons in regard to cost of the tanning material in the acceleration of the tanning and in the good yield.

Use of Extracts for Tanning in the Pit.—The better qualities of extracts may be utilised with moderation in the first part of the work. However, their use does not commence until the rehandling or the steeping in the pit. To use extract in the rehandling commence with pine bark liquor, then add each week a small quantity of 25° extract, gradually increasing the strength from 15 tenths by the tannin indicator up to 25 or 30 tenths ($2\frac{1}{2}$° to 3° B.). If it be not desired to use the extracts until after steeping in the pit the skins are spread out in the usual way with good bark suitably ground so as not to make a mortar and to

enable the juice to circulate ; they are then drenched with a mixture of extracts and weak liquor.

Method of Working.—Drench very liberally with liquor consisting of a mixture of extract and weak liquor taken from the liquor pits ; bring this mixture to 20 to 25 tenths on the tannin indicator for the first powder ; from 25 to 30 tenths for the second, and 30 to 35 tenths for the third, if three powders be given, as in the case of heavy leather or ox-hide for belting. Take care at the outset to fix a channel in the pit, by which the liquor can be drawn off when desired, either to replace it by other liquor or simply to displace it by pumping the pit on itself. After two, three or four months (give the powders every two months at the farthest) the pit is turned or dry piled, then the liquor is withdrawn by the pump, or in any other way, in proportion as the skins are lifted and run to the liquor pits. The spent tan is also carried there. It will consequently be seen that in a very short time the liquor pits have become very rich. Instead of having on the front pits liquor weighing at the most 10 tenths, as when bark is used alone, the liquor will very soon test 20 to 25 in the front pit, and in the tail pit, if the exhaustion has been done methodically, it will test 0 or very nearly so. What is to be done with this strong liquor? It can be used to form the bath in the handling which immediately precedes the laying in the pit. This bath is formed at 20° or 25°, according to the strength of the liquor. In the preceding rehandling, or in the vat, if only one handling be done, the bath is formed with liquor from the second liquor pit, which will test 16 to 18 tenths, then in the following pits there will be liquor at 14, 12, 10, 8, 5, which will be used in the first vats ; the weakest liquors, when vatted, become stronger and stronger as the skins age in manufacture. By this method of working the pits an important economy in time may be realised, either because only two pits are given instead of three, or because the duration of each is abbreviated. By using 25° chestnut extract, simply clarified for tanning, ordinary coloured leather is obtained ; by using clarified chestnut extract, rendered soluble, brighter coloured leathers are obtained.

Tanning of Leather ("en croûte") to be Tanned and Dried but not Curried.—Extracts soluble in the cold are employed with barks titrating 10 tenths (1° B.) to drench the pits, or for the initial bath (tanning by immersion), so as to increase the daily strength by 2 tenths of a degree, by small additions of extract, so that in fifteen days a bath of 40 tenths (4° B.), which gradually penetrates and passes through the leather *en croûte*, without either rendering it brittle or shrivelling it. The rapidity of tanning can evidently be accelerated

by using a heating coil in the bath in which warm water of 30° to 35° C. circulates. This mild heat facilitates the assimilation of the tannin by the leather, swelling the skin, and producing *croûtes*, soft and pliant to the touch. The addition of extract to the tanning baths or pits thus produces white *croûtes* of high yield, provided, however, that they be dried in the dark as far as possible. The manipulation of the *croûtes* during tanning can only be guided by practice and the routine of tanning adopted in each factory, the principles and the manner of working of which are different.

Dissolving the Extracts.—It is of the greatest importance that the extract to be used in tanning be uniformly distributed throughout the liquor to which it is added, because the extract, however pure and however soluble it may be in the cold, may remain imperfectly dissolved if the precautions incidental to its use be not taken. The tanner thus suffers a loss and irregularity occurs in the results of his operations. The extract ought always to be added to the upper surface of the water or liquor of the vessel or pit, and not to the bottom, that is to say, the extract is to be run into the liquor or juice slowly and not in a gush, taking care to stir constantly. The desired quantity of extract should never be discharged into the bottom of the reservoir or mixing vat, for water to be added subsequently on the top. It would be in vain to stir afterwards; complete solution would not be obtained. The best method of working is to heat in a vat to 30° to 35° C. (86° to 95° F.) (or better, use condensed water) a quantity of water at least equal to that of the extract to be used; then to pour the extract thereon, slowly stirring, as described above. This first solution, which works very well owing to the favourable temperature, ensures the perfect utilisation of the extract.

History of Rapid Tanning.—The idea of rapid tanning has had admirers in all ages, and has been the object of many researches; many tanners have dedicated thereto their knowledge, their time and their money. Without reckoning the well-known names of Seguin, of Knoderer, of Worms and Balé, there is not a tanner, we believe, who has not made experiments to reduce, more or less, the long time occupied in tanning. Some Italian tanners in the time of the Italian wars tanned more or less rapidly, by using the "tumbler" even at that early date. It must be added, however, that the leathers so tanned were accepted as meeting the urgent exigencies of the moment, and that slow tanning was perforce returned to as soon as urgent necessity had passed away. The system of Fratelli Durio (the famous tanners of Turin, who kindly enabled the authors to inspect their important and improved tannery during the Congress of Leather

Chemists) is certainly the result of a series of profound and rational researches on all which had been done previously, a new milestone placed after other milestones on the road of progress of rapid tanning, but it may be also asserted that it is to the initiative of these tanners themselves that the true theory of rapid tanning is due, and its definite adoption into actual practice. In 1881 they wished to buy the Italian rights of the Worms & Balé patent, based on electricity, and before entering into further negotiations, they desired to make inquiries on the industrial value of that process, but after some experiments, they concluded that the famous electric fluid exercised no influence on tanning. This patent did not, therefore, present any rational guarantee, even as regards the very incomplete results yielded by the said process. Moreover, it is well known that all the tanneries which tested it at the time had to give it up very soon, and the firm of Worms & Balé itself had finally to resort to the system of Fratelli Durio, the principle of which was antecedent to their own, because in fact the so-called electric action on the leather was no other than the effect of the rotation or fall which took place in the fulling machines of the Turin tanners. It was, therefore, whilst pursuing experiments on extract and on the tumbler that Messrs. Jacques and Segondo Durio, in 1882, almost on the point of being disheartened, finally solved the question of rapid tanning at high strength: the old theory of time and tan had to disappear from that day. Their idea was contested in the tanning world; then being forced to recognise its value, they tried to diminish it by attributing the invention to Knoderer (1850), asserting that if he had had extracts at his disposal, he would have tanned as well as Fratelli Durio. To make this assertion is not to know the tenor of Knoderer's patent, which says that this inventor only used non-concentrated, therefore weak liquors, and that he, moreover, prescribed that the work should be done gradually, so as not to attack the skins too strongly at their exit from the river treatment. The system of Fratelli Durio is applicable to all sorts of skins and for all purposes: sole leather (smooth), upper leather, belts, saddlery. Any desired shade may be obtained at will. The different types of leathers produced and asked for by customers in different countries are very easily matched. The tanning is perfect and compact, the flower always soft and fine, finally the specific gravity, tanning being more complete, is higher than in ordinary tanning. Like all patents, that of Fratelli Durio has had its improvers and its professors, who have hastily produced different processes, by the addition of foreign matters to the extracts, or who have asked the tanners to work *in vacuo* or under pressure. They have all met the fate which

might be expected: they were still-born. Only the system of Fratelli Durio has continued to furnish to the whole world its indications of principle and simplicity, which have alone contributed to its success and which will impel its adoption by force of circumstance: other times, other manners. It is absolutely certain that if 20 per cent. of the tanners tanned with more rapid processes a consumption of extract would ensue, which would render it necessary to double the extract factories. That would be a real revolution in the tannery and an evolution of the tannin industry, which, moreover, only seeks to develop itself farther, provided that it is possible to find in India, in Australia, in Africa, in South America and in the United States various tanning materials capable of supplying it for an almost indefinite number of years. This appears quite possible when we reflect that the methodical exploitation of different woods may be renewed at intervals, which would counteract the deafforestation already spoken of in France, including Corsica.

Ultra-rapid Tanning.—Into a rotary wooden "tumbler," such as is made by the tannery machine constructors, of 10,000 litres (2,200 gallons) capacity, mounted on a hollow spindle with wooden blades in the interior, and driven by an alternate motion with a speed of ten turns a minute, the skins to be tanned are introduced through a bronze manhole. In the case of small skins, the tannic liquor contains 3 per cent. of tannin; it is generally made by dissolving mixed chestnut and quebracho or mimosa extract in water; for heavy leathers, the tannic liquor contains 5 to 8 per cent. To facilitate the penetration of the tannin into these heavy leathers they are first subjected to a radical treatment in river water, followed by a swelling bath, consisting of bark liquor, to which formic acid has been added in the proportion of 200 grammes per 100 kilogrammes of leather in bands. To neutralise the astringency of the chestnut tannin, it is well to use along with this extract some quebracho or mimosa, in proportions which vary with the origin and the nature of the skins to be tanned. In all cases the tanning of heavy leathers may be accomplished in forty-eight or sixty hours, that of small skins in eighteen or twenty-four hours. Each fulling tumbler may tan about 50 tons of leather for belts or soles yearly; 12 to 15 square metres of surface space suffice for its installation. It requires 2 h.-p. This process, which does away with the pits, tan mills, sheds and stitchers, with an expenditure of 1·8 to 2 kilogrammes of extract of 25° per kilogramme of finished leather, gives a 55 per cent. yield, and the cost of tanning a kilogramme of finished leather only amounts to ·6 franc (say 2½d.) per pound. In order that the skins may be regularly tanned the liquor must be maintained at constant density. This result

is obtained by making a communication between one of the hollow spindles of the tumblers and an elevated reservoir in which the extracts are mixed with water. After some successive operations the liquor from the tumbler ought to be evacuated and clarified before being used again with new liquors.

CHAPTER VI

MANUFACTURE AND USE OF OAK EXTRACT

Oak Wood—Barks—Equipment and Plant of an Extract Factory—Method of Manufacture according to Albert Thompson and Emile Blin's Patent

Oak Wood.—The oak (*Quercus*), the bark of which is *the* tanning material *par excellence*, supplies a wood containing 5 per cent. of tannin. Hartig found that the young wood, cut in the beginning of May, contains from 4 to 7 per cent. of tannin, whilst in winter this rises to 10 per cent. of the dry matter. The same author found, in the heart-wood of oak 160 years old, 12 to 14 per cent. of tannin. The percentage of tannin in the wood is maintained if, after felling, the wood is barked and sawn up; after several months the unbarked wood diminishes in strength, from the centre to the circumference. The alburnum contains little tannin; the lower parts of the tree contain the most. According to the experiments of Professor Henry of the French National School of Forestry, the following conclusions may be drawn: (1) The percentage of tannin progressively diminishes both in the bark and in the wood from the bottom to the top of the trunk, at least in the case of *Quercus robur*. (2) On a cross section it is always the alburnum which contains the least, generally from 1 to 3 per cent., then suddenly the tannin attains its maximum development in the peripheric layers of the duramen (6 to 10 per cent. in the oak, 13 to 15 per cent. in the chestnut), and continues to diminish, more or less regularly, to the centre. The large branches behave like the trunk. (3) Everything else being equal, the wood of an oak or a chestnut will be richer in tannin as its *cime* is more ample, more isolated, better illuminated, or in other words as its annual layers are wider. (4) A circular slice, exposed for a year to the weather, loses three-fourths of its tannin in its bark and alburnum, the half only of that in the wood. This difference is explained by the fact, that in the bark and in the alburnum the tannin is dissolved in the *lumen* [albumen ?] of the cells, whilst in the duramen it impregnates the walls of all the tissues so intimately that a series of macera-

tions on the water bath, followed by several pressings, is necessary to extract it. (5) It has been demonstrated that under the action of oxygen or of fungi such as the *polyporus sulphureus et igniarius*, which induce first a red rot, the second a white rot in oak, the wood loses its tannin, whilst it preserves indefinitely a notable proportion of this principle, so unstable, however, when these two causes of decay are eliminated. An enormous oak trunk of the quaternary epoch embedded in the soil of Nancy still contained 2·36 per cent. of tannin.

Varieties.—Only the principal species yielding the different barks used in tanning or in the tannin industry, *Quercus robur, Quercus pedunculata, Quercus ilex, Quercus rubra,* will be described. The evergreen oak (*Quercus ilex* L.), the bark of which is used for tanning light leathers. The white oak (*Quercus sessiliflor* Smith) and its tropical variety *Quercus pubescens,* the bark of which is specially used for tanning heavy sole leather. This bark gives a yield of 28 per cent. of 25° extract. It therefore takes 350 kilogrammes of this bark to produce 100 kilogrammes of 25° extract. The zeen oak (*Quercus mirbeckû* Duvieux). The kermes oak (*Quercus coccifera* L.), the roots of which yield *la garouille* which is used in tanning heavy leathers intended for belts. The cork oak (*Quercus suber* L.) (*Quercus occidentalis* Gay, in the Landes).

Percentage Strength of Tannin in some Varieties.—White oak of La Drôme (Puygiron, near Monte Limar). These oaks of twenty to twenty-five years old, only beginning to form perfect wood or duramen, give the following percentages of tannin in different organs on analysis (see A in following table):—

TABLE XXV.—DISTRIBUTION OF TANNIN IN DIFFERENT PARTS OF THE WOOD OF DIFFERENT VARIETIES OF THE OAK

	A.	B.	C.
Bark dried at 100° C.	10·8	8·53	7·74
Alburnum external	2·2	1·95	1·98
„ internal	1·7	3·18	1·74
Duramen external	...	15·60	9·68
„ internal	...	11·08	5·87

B, *chêne* (oak) *tauzin* d'Angers; C, *chêne* (oak) *tauzin* de Mauléon.

The percentage of tannin in the bark, whether of white oak or of evergreen oak, always varies essentially with the source, but there is an especially propitious moment for collecting it, which affects this

percentage, *i.e.*, as soon as it is packed in bags it must be protected from rain and intemperate weather which prevails during its collection. Tanners are especially interested in selecting this propitious moment for receiving their annual stocks of different barks.

Manufacture of Extract.—Oak extract is manufactured in the same way as chestnut extract, except that the maceration of the chips in open vats is obligatory, and their clarification should be pushed to the extremity, owing to the more intense dark colour of the liquors. In France this manufacture is of little importance, looking to the small number of hectares at disposal for this exploitation, and especially to the poor yield of this wood, which does not exceed in a factory, working according to the latest improvements, 14 per cent. Certain factories use the top branches of oak which barely yield 7 per cent. with a 25° extract containing 22 to 25 per cent. of tannin. Other manufacturers only use for the manufacture of oak extract the blocks and the large branches of unbarked oak, of about 20 cm. in diameter, or the trunk split into logs of 50 to 60 kilogrammes (110 to 132 lb.). There is no profit in extracting the tannin from branches of 2 to 3 cm. in diameter, because, in the first place, they are too poor in that principle (2·2 to 2·5 per cent. of tannin), and in the second place, because they have an appreciable value as wood for charcoal. The twigs contain 4·7 to 6·6 per cent. of tannin. It would therefore be desirable for a wood distillery situated near cheap transport routes (canals) to add an extract factory, which would utilise, for its purposes, the oak brushwood, the value of which would enable the proprietors to realise a great economy on the purchase of their raw materials. Oak extract, employed in tanning as an adjunct, imparts to the leather a similar colour to that of bark-tanned leather; moreover, it may be used in larger proportion than chestnut extract, because it contains a host of products which have the most beneficial effect on the suppleness or pliancy of the leather (amylaceous, mucilaginous matter, etc.), which enable it to be used, not only in the tanning of sole leather (for which it is moreover employed) but also in the tanning of leather for uppers and other light leathers. The cost of oak extract, being about 20 francs per 100 kilogrammes (8s. per cwt.), it will be seen that its sale is, consequently, very limited. Thus the authors only know of two factories actually making pure oak extract. The Hungarian and Russian extracts reach France at a more rational price. As a brand of pure oak extract, the authors would quote that made by the oak extract company of Russia, whose works are situated at Kurenewka, near to Kieff. That factory, which dates from 1902, produces annually 6,000 tons of liquid extract guaranteed to contain

27 per cent. of assimilable tannin. The average composition of these extracts is as follows :—

TABLE XXVI.—SHOWING THE COMPOSITION OF RUSSIAN BRAND OF PURE OAK EXTRACT

	Per cent.
Soluble tannin matter	3·07
Non-tannins	10·9
Water	58
Insoluble	0·4=100°

Degree Beaumé at 18° C., 25·8. The authors may also quote as a genuine oak extract that manufactured by Miller's Standard Extract Co. at their Slavonia factory (exported through Fiume).

Plant and Equipment of an Oak Extract Factory.—The following is a specification for a factory of this nature, the basis of production of which is 4½ metric tons of 25° extract, and which works according to a patent described farther on. Two boilers with a heating surface of 140 square metres, gauged to six atmospheres ; one Laboulais horizontal motor of 85 h.-p., 90 revolutions ; one horizontal motor of 10 h.-p., 200 revolutions ; one dynamo 110 volts, 30 ampères ; table for distributing the electric light installation, comprising two arc lamps and thirty-eight lamps of 16 candle-power ; belts ; transmission shafting ; pulleys ; plummer blocks ; gearing ; lubricators ; one cutter, 20 tons output ; one cutter, 30 tons output ; one cup elevator ; eight vats for wood diffusion, each of 9,000 litres (1,980 gallons) capacity ; nine vats for various manipulations of the liquor of 10,000, 4,000 and 2,000 litres (2,200, 880 and 440 gallons) capacity ; one filter press, Lumpp system ; two filter presses with pumps and their respective motors, Johnson's system ; two sets of copper concentration plant, Gouyer's system, of 70 square metres of heating surface ; one copper vat of 1,200 litres (264 gallons) capacity for the preparation of decolorisers ; one wrought-iron hot water tank of 7,300 litres (1,606 gallons) capacity ; one Lawrence copper refrigerating system ; three wooden vats ; one condensation pump, 25 cm. (25 tons) capacity per hour ; one elevation pump, 25 cm. capacity per hour ; one feed pump, 3,000 litres (660 gallons) capacity per hour ; one centrifugal pump, 6,000 litres capacity per hour ; one air pump and condenser, Worthington system ; two sandstone grinding stones on cast-iron foundation for sharpening the cutters ; one emery grinding stone on cast-iron foundation ; iron, copper, and lead piping ; copper taps and fittings ; 300 metres of Décauville rails with six turn-tables, points ; four tilting trucks with skips ; three wood waggons ; one wood barge of 15 tons ; one Décauville crane on wooden staging for discharging vessels ; one weighing machine of 10 metric tons load.

The following is given *in extenso* as a matter of curiosity. *Descriptive Memoir Lodged in Support of the Application for a Fifteen Years' Patent for Improvements in the Manufacture of Tannic Liquors by Albert Thompson and Emile Blin.* The invention, which is the subject of the present application, relates to the manufacture of tanning liquors used in the tanning of skins for the preparation of leather.

The tanning liquors, generally obtained by diffusion in boiling water of certain woods rich in tannin, previously crushed to facilitate the extraction of the tannic principles which they contain, are commonly withdrawn from the diffusion vats when they reach 3° to 4° Beaumé. These liquors are then clarified by means of substances amongst which acids (sulphuric acid, oxalic acid, etc.) intervene so as to precipitate or destroy the salts and coloured organic matter which they contain. This clarification effected, the liquors are then filtered to free them from the deposits induced by clarification, then they are finally concentrated in evaporation plant up to a density of 20°, 25°, 30° Beaumé and more, according to the requirements of consumers. The tannic liquors, treated in this way by acids, always remain acid, and are impoverished in very appreciable proportion of their useful principles. Moreover, the oleaginous products employed in the majority of acid processes, to attenuate the destructive effect of these acids and to reimpart to the tannic liquors the velvety lustre which the latter remove, present many disadvantages, especially those which consist in forming spots, and in falsifying the density of the tannic liquor properly so called. Finally, the decolorisation of the tannic liquors, limited perforce by the loss of useful principles, due to the adoption of acid processes, has not as yet allowed tannic extracts of certain categories to be utilised, hence for the working of very light and very pale leathers there are not to be found at the present time tannic liquors sufficiently decolorised and of sufficient strength to be used for that kind of work. In our invention the tannic liquors, always obtained by diffusion, are clarified and decolorised without the use of acid or oleaginous product, and contain in proportion more utilisable principles than the tannic liquors treated by acid processes. They are neither acid, nor do they cause spots, and they contain no body capable of altering the natural density. Moreover, as great a decolorisation as consumers can desire is obtained by our process, and that without appreciably diminishing the percentage of useful principles. They possess indisputable qualities from the point of view of tanning leather. All these results being obtained without destroying the natural velvety lustre of the tannic liquors and without resource to the use of acids. Our invention rests on the following researches: mineral acids, sulphuric acid, for example, precipi-

tate lime salts, and destroy certain organic principles present in the tannic liquors, and thus decolorise them. But these acids have a deadly effect on the tannin, and cause its energetic oxidation, especially when warm, and an appreciable quantity of tannic acid is transformed by concentration into gallic acid. Now it must not be forgotten that the products of oxidation of physiological tannin neither precipitate gelatine nor albumen. They are therefore useless. On the other hand, they give black colorations with salts of copper and salts of iron, and are therefore injurious. A series of methodical researches has caused us to resort to the use of vegetable alkaloids, preferably the vegetable bases naturally contained in quinquinas, quinine, quinidine, cinchonine, cinchonidine and their isomers or derivatives. We use either one of the alkaloids separately, or a mixture of two or more bases as need be. (1) These bases, free or in the condition of salts, are precipitated by pure physiological tannin, and the precipitate of tannate is soluble in an excess of tannin or organic acid. (2) In tannic liquors the precipitates formed by these bases, or their salts, do not redissolve in an excess of liquor. It was, therefore, to be foreseen that decolorisation, produced by this method, would precipitate but very little pure tannin. Experience fully confirmed the fact, and our liquors are much more rich in tannin material than those clarified by other processes. Besides they are in no way damaged by the decolorisation, and retain their natural velvety lustre, so much sought after by tanners. Finally, the greater part of the vegetable alkaloids being found in the deposit from the clarification, we recover them by one of the processes used in the extraction of quinquina. We applied the following process: The drained precipitate is treated warm by water acidulated to the extent of 5 parts in 1,000 by hydrochloric acid. The liquor is filtered and treated by milk of lime, gauged in the proportion of 10 grammes of quicklime per litre of hydrochloric liquor. The alkaloids are precipitated by the excess of lime. The precipitate is collected, washed with cold water, then treated by a slight excess of water acidulated by sulphuric acid. The acid liquor containing the alkaloids is filtered, decolorised warm by animal black, again filtered and neutralised by quinine or one of the other alkaloids precipitated by carbonate of soda in a portion of the liquor. It only remains to titrate the solution obtained. It is to be understood that we reserve the right of applying such process of extraction as we may desire, having only pointed out the above process to show the economy of our invention from a commercial point of view.

Method of Working.—The tannic liquor as it comes from the diffusion vat has a density varying commercially from 3° to 4° Beaumé. (The

strength is obtained naturally as desired.) It is allowed to cool to the surrounding temperature; when cool an aqueous solution of the alkaloidal salts of quinquina or of these pure alkaloids, as occasion may require, is run into the tannic liquor, stirring energetically for a few minutes (five minutes suffice). The salts of the alkaloids enter into this operation in the proportion of 1, 2 or 3 kilogrammes per metric ton of extract brought to 20° Beaumé, according to the decolorisation which it is desired to obtain. It is evident that less or greater quantities than those indicated may be used according to the result which it is desired to obtain. Under the action of these alkaloids the coloured or extraneous matters in the tannin, as well as a certain quantity of the latter, are precipitated along with the alkaloids, etc., leaving the liquor with its varnished appearance, so much sought after by consumers. We ought to say that the same results are obtained, whatever the temperature. But as warm working operates much more slowly, this method appears to us economically less practical, and that is why we have preferably indicated cold treatment in the first place, which is more rapid the lower the temperature. We may recall that in our process no dangerous caustic or poisonous substance is used and that we cause the greater part of the alkaloids used to re-enter into the manufacture.

Résumé.—Summing up, we claim as our invention and our exclusive property: (1) The process which consists in using quinine alkaloids, either mixed or separately, for the clarification and decolorisation of tannic liquors obtained by diffusion. The said alkaloids pure or in the state of saline combination used in proportion varying with the decolorisation which it is desired to obtain, but in sufficient quantity, according to us, in a maximum proportion of 4 kilogrammes per metric ton of liquor brought to 20° Beaumé. This proportion enabling almost white tannic extracts to be obtained and used indifferently, hot or cold, without distinction of degree of temperature, the said process having for its object the precipitation without the aid of an acid of the coloured or foreign substances which may exist in tannic liquors, whilst at the same time preserving to these latter, without the aid of any oleaginous or other greasy body, the varnish-like appearance which it ought to have for consumption and which it naturally possesses. (2) As a new commercial product the tannic liquor presenting the following characteristics: free from acid exempt from any oleaginous or greasy body, preserving naturally its velvety and varnish-like appearance, possessing, notwithstanding its decolorisation having been pushed to an extreme point, a percentage of tannin higher than that obtained with acid processes, the said product enabling the tanning of skins to be effected under better conditions and extending its use to certain classes of leather up to now

deprived of this style of tanning. Moreover, this tannic liquor possesses as an essential quality the characteristic that it is less sensitive to the oxidising influences of the air than that obtained by acid processes, which always contain gallic acid destitute of tanning power, neither precipitating gelatine nor albumen, and injurious because they give dark colorations with metallic salts.

For THOMPSON and BLIN,
(Signed) ARMENGAUD aîné.

Seen to be annexed to the patent of fifteen years taken 28th July, 1899, by Thompson and Blin. Paris, 13th November, 1899.

CHAPTER VII

MANUFACTURE AND USE OF QUEBRACHO EXTRACT

Quebracho Wood. Generalities.—Quebracho (*Aspidospermum quebracho*) is a tree of the family of Apocynaceæ growing in the Argentine Republic, Uruguay, Brazil, Guiana, but it is especially in Paraguay where its exploitation is at present carried on (several factories have been installed there since 1902), as well as in the Argentine Republic to the north of Santa Fé, on the Rio Parana, and at Véra, which is 250 kilometres from Santa Fé, where there are important exploitations of quebracho colorado for the manufacture of dry extracts on the spot. Its importation into France dates from 1873. It is a very durable, hard, red wood of average elasticity, even slightly brittle. Its density is 1·250. There are two varieties of quebracho, colorado and blancho (red and white); the quebracho colorado contains 16 to 19 per cent. of tannin and the quebracho blancho from 12 to 13 per cent. On analysis it yields:—

TABLE XXVII.—SHOWING THE PROXIMATE COMPOSITION OF QUEBRACHO WOOD

	Per cent.
Water	14
Tannin calculated as dry matter	20
Extractive matter	10
Woody fibre	55

Quebracho contains, beside extractive matters, colouring principles difficult to eliminate, and which have greatly hindered its use, because they impart to the leather an unpleasant red colour, but which at the present day are removed by chemical treatment which clarifies and decolorises them. The bark of the wood does not tan leather; it only contains 1·55 to 2·25 per cent. of tannin (Villon). This bark has often been confused with that from the curupay (*Acacia curupy*). Other woods resemble quebracho in hardness and in appearance, but do not contain tannin. The tan wood called tanho (*Aspidospermum churneum*) contains 7 per cent. of tannin; the peroba (*A. peroba*) 5 per cent., the peyma amarella (*A. sessiflora*) 10 per cent., the fever bark 4 per cent.

Manufacture.—Quebracho extract is made in the same way as chestnut or oak extracts. The wood is cut into chips, then macerated in wooden vats in the open air or in autoclaves. The quebracho liquors so obtained are clarified and concentrated by triple effect to 25° or 30° B. or evaporated to dryness. In normal manufacture, and according to the kind of quebracho wood treated, the yield in 25° extract is from 38 to 45 per cent. of the wood treated. The disadvantage under which the French manufacturers of this extract are now working at the present time as compared with the tropical countries will be understood, especially since the South American factories work normally, if we bear in mind that the average price per ton of quebracho wood at Havre fluctuates between 115 and 117 francs (£4 12s. to £4 13s. 7d.) delivered (constantly rising). If there be added to the cost of the raw materials to manufacture 100 kilogrammes of extract at 25° (it takes 250 kilogrammes of wood) a minimum of 5 francs for working expenses, special allowance being made for the difficulty of cutting and handling that wood, it will be seen that there is no advantage in manufacturing quebracho extract in France, and that tanners would do better to purchase for their use dry Argentine extract or some other brand, and to prepare for themselves a liquid 25° extract with a net profit of 4 francs per 100 kilogrammes. The cost price of French manufacturers cannot be materially lowered, whilst that of the tropical extractors will undoubtedly fall through over-production. Besides, the purity and the percentage of tannin in these South American extracts are now well known. It will suffice to give here the composition of known brands.

TABLE XXVIII.—SHOWING THE PERCENTAGE COMPOSITION OF QUEBRACHO WOOD EXTRACT

	A.	B.	C.	D.	E.
	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.
Tannin matter . . .	64·9	69·60	64·8	45·65	31·4
Non-tannins . . .	4·9	9·60	16·4	5·05	9·4
Water	13·5	13·12	17·7	46·70	56·0
Insoluble	16·7	7·68	1·1	2·60	3·2
Total . . .	100·0	100·00	100·0	100·00	100·0

A, Harteneck Hermanos of Buenos Ayres; B, Casada; C, G. B. M. decolorised; D, Pasty extract decolorised; E, Average composition of liquid 25° extract of French manufacture.

The authors have made numerous tests with 25° quebracho extract, using dry extracts of South American origin, using the same amount

of alkaline sulphites as that indicated by the composition of a 25° French extract. They have practically demonstrated that 100 kilogrammes of dry extract yield a minimum of 225 kilogrammes of 25° extract, completely soluble in the cold and quite assimilable by the leather, whilst yielding quite as pretty a tint as any decolorised.

Results of the Authors' Sulphitation Experiments.—The following are the results obtained by simply varying the proportion of alkaline sulphites used per 100 kilogrammes of 25° extract :—

TABLE XXIX.—SHOWING RESULTS OF EXPERIMENTS ON THE SULPHITATION QUEBRACHO EXTRACT

Sulphite per cent.	11.	13.	15.	18.	5 to 20.
Tannin matter	38·2	37·8	36·6	34·8	32·0
Non-tannins	6·2	6·4	7·8	8·2	10·6
Water	55·6	55·8	57·0	57·0	57·4
Insoluble	0·0	0·0	0·0	0·0	0·0
Total	100·0	100·0	100·0	100·0	100·0
Total soluble per cent.	44·4	44·2	44·4	43·0	42·6
Yield per cent. in 25° extract . . .	180·0	185·0	200·0	204·0	210·0

It will be seen that the percentage of "soluble" does not vary more than 2 per cent. from one experiment to another, and that in spite of the increase in the addition of sulphite, only the percentage of tannin decreases. The tanner, therefore, has no interest in an extract sulphited in the ratio of 20 to 25 per cent., but rather in an extract sulphited to a minimum extent, *i.e.*, containing a maximum of soluble tannin. Now, as that sulphited to the extent of 11 per cent. contains no insoluble, its use is quite demonstrated in good tanning, which should exclude a large quantity of sulphites, as being liable to injure the quality of the leather, owing to the difficulty in eliminating it. Moreover, 25° quebracho extract, sulphited only to the extent of 5 per cent., produces leathers with a greater yield than those tanned with extracts sulphited to a greater extent, whilst still retaining much greater suppleness (pliancy). This class of extract is made directly in a single vessel (Fig. 83), by running in the dry extract[1] all at once, and the right amount of water; the mass is then heated for some hours, after which the corresponding amount of 25° extract (*premier jet*) and perfectly soluble in the cold is run off.

[1] There are now on the foreign (to France) market brands of soluble quebracho containing only about 5 per cent. (ideal) of sulphite and sufficiently decolorised to be used directly in the tannery.

As a matter of interest, there is here given a copy of a patent (*brevet*) for fifteen years for a "Process of converting dyewood and tanning extracts into new products of greater industrial value by the action of the bisulphites, sulphites, and hydrosulphites of soda potash," etc. Application made by La Société Lepetit, Dollfus et Gansser.

Descriptive Memorandum.—Of late years the action of alkaline sulphites on bodies of the aromatic series has been repeatedly investigated. We quote as examples the German patents 56,058 and 76,458 of Fischesser (Wagner's *Jahresb. der Chemischen Industrie*, 1895, p. 602), the German patents 86,097 of 18th February, 1895 ; an application for patent of the Farbwerke Hoechsten, 1895, according to which a reaction, previously investigated by Piria, is applied (Liebig's *Annalen*, v., 78, p. 31), *viz.*, the action of sulphites on nitro-naphthalene. We have investigated the action of sulphites and bisulphites on dyewood extracts, *e.g.*, yellow wood, fustic, quercitron extracts and on tannin extracts, *e.g.*, sumac, quebracho, chestnut, gambier, etc., and we have found that under certain conditions new products are formed, which differ essentially from those employed as raw material and are of great commercial value. These new products and their manufacture are the object of the present application for patent, and we explain our process by the following examples :—

Example I. Preparation of a New Colouring Substance by the Action of Bisulphite of Soda on Yellow Wood Extract.—We heat in an autoclave, at a temperature of 110° to 115° C., for eight hours, 300 kilogrammes of 28° yellow wood extract and 100 kilogrammes of sodium bisulphite of 35° B. ; the yellow extract is then converted into a pale yellow, very dense mass, which has the appearance of pure *morin ;* it consists of microscopic needles, difficultly soluble even in hot water and representing an addition product similar to the addition compounds which aldehydes give with bisulphites. Acids easily liberate sulphurous acid from it, but do not, however, regenerate yellow extract with all its properties. The product so obtained, used such as it is, or neutralised by carbonate of soda, or well purified by washing with cold water, dyes cotton and wool on mordants, imparting thereto much brighter, deeper and more pure tints than Cuba extracts ; it gives pure yellow tones on alumina in calico printing.

Example II. Preparation of a New Colouring Substance by the Action of Bisulphite of Soda on Fustic Extract.—We heat in an autoclave, at a temperature of 145° to 150° C., for eight hours, 300 kilogrammes of 28° B. fustic extract and 100 kilogrammes of bisulphite of soda of 35° B. The fustic extract is converted into a dense mass of a very pale, greyish yellow, almost white when dry, very slightly soluble in hot water,

soluble to a fine bright yellow in alkalis. The product differs from the fustic extract used, in the same way as the derivative of the yellow wood extract differs from the extract itself, *i.e.*, it yields on wool and cotton dyeing it on mordants much purer and much brighter tints.

Example III. Conversion of Quercitron Extract into an Easy Soluble Dye of Greater Intensity.—We heat in an autoclave, to a temperature of about 150° C., from six to eight hours, 300 kilogrammes of 25° B. quercitron extract and 100 kilogrammes of bisulphite of soda of 35° B. The product obtained differs from the pure quercitron used, in so far that it dissolves with the greatest facility in cold water, a very desirable property in calico printing, and by the fact that it possesses a much more intensive colouring power. The action of the bisulphite, in this example, has a certain analogy with the action of dilute acids on quercitron extract, so as to decompose them into sugar and colouring principles, but the decomposition of glucoside by boiling dilute acids being accompanied by the formation of secondary bodies, the product so obtained has not the purity of tone which can be obtained by means of bisulphite.

Example IV. Conversion of Quebracho Extracts, generally Imperfectly Soluble in Water, into a Tannin Extract Completely Soluble in Cold Water.—(*a*) We heat in an autoclave, for six hours, at 140° to 150° C., 300 kilogrammes of quebracho extract of 25° B., and 80 kilogrammes of bisulphite of soda of 35° B.

(*b*) We heat in a jacketed pan, fitted with an agitator and heated by steam of one and a half to three atmospheres, for eight to ten hours, with constant agitation, 300 kilogrammes of quebracho extract and 100 kilogrammes of bisulphite of soda, or of neutral sulphite of soda of 30° B., or 130 kilogrammes of hydrosulphite of soda of 20° B.

(*c*) Instead of converting quebracho extract into a soluble product, the latter may be obtained directly by extracting quebracho wood, with or without pressure, adding for every 100 kilogrammes of wood 12 to 15 kilogrammes of bisulphite or sulphite of soda to the water used to extract the wood. The extract obtained according to (*a*), (*b*), (*c*) presents the same properties: it is more fluid than the present commercial quebracho extracts, dissolves easily in cold water without producing any insoluble deposit, it tans skin rapidly and imparts to it slightly rose tints, recalling those produced by mimosa bark, so highly prized by connoisseurs; moreover, the leather has a remarkable suppleness (pliancy) which is not obtained with ordinary quebracho. Naturally the time, the temperature and the proportions given in the present application for patent vary within rather wide limits. Instead of bisulphite of soda those of ammonia and potash may be used. The

application to Persian berries, wood, hemlock, catechu, *pigou*, etc., is the same as that described in the examples quoted. Summing up, we claim as our exclusive property: (1) The process consisting in heating dyewood extracts or extracts for tanneries, such as yellow wood, fustic, quercitron, quebracho, hemlock, chestnut, etc., extracts, with sulphurous acid, alkaline bisulphites, sulphites and hydrosulphites, in an autoclave to temperatures higher than 100° C. or in the open air. (2) The products obtained by this process. (3) The application of these products to dyeing and calico printing and tanning or to the preparation of new dyes.

Copy of the Certificate of Addition to the Patent for Fifteen Years, 20th April, 1896, No. 255,698, for "A process of converting dyewood and tannin extracts into new products of greater commercial value, by the action of the bisulphites, sulphites or hydrosulphites of soda, potash," etc. Application made by La Société Lepetit, Dollfus et Gansser.

Descriptive Memorandum.—In our chief patent of 20th April, 1896, No. 255,698, we described the preparation of new tinctorial products, bisulphite compounds of the colouring principles of yellow wood and fustic, and also the conversion of ordinary quebracho, hemlock and pine extracts, soluble with difficulty in the cold, into easily soluble extracts, and we mentioned the altogether special importance of this slightly soluble extract for the different purposes of rapid tanning. In continuing our experiments on the preparation of these extracts, we have found that they are rendered soluble by the fact that in heating quebracho and hemlock extracts with bisulphites and sulphites for a somewhat long time, a portion of the sulphurous acid being expelled, neutral sulphites with an alkaline reaction are formed, which facilitate the solution of the resinous principles of the extracts. The excess of sulphurous acid in contact with the extract during the period of treatment prevents any oxidation, and induces at the same time a certain decolorisation of the extract, owing to its reducing action. We have found that soluble extracts are also obtained when alkaline bisulphites, sulphites or hydrosulphites are wholly or partially replaced by some alkaline salts of sodium, potassium or ammonium, such as borates, carbonates, bicarbonates, phosphates, lactates, or sulphides, or hydroxides of sodium, potassium or ammonium. It is always necessary to render the extracts soluble by working in the hot condition. But the operation is a very short one (stead of lasting seven to eight hours in the conditions described in our chief patent), and requires a smaller quantity of added products for the conversion into soluble extracts. It is always advisable to add to the products, which render the extracts soluble, a certain quantity of reducing substances, such as

sulphites, bisulphites, hydrosulphites or sulphides, so as to prevent any oxidation; but such procedure is not absolutely necessary.

Example.—(*a*) 1,000 kilogrammes of 26° B. quebracho extract are heated in a steam-jacketed pan fitted with an agitator up to 90° or 100° C., and ammonia, diluted with its own weight of water (it takes about 76 kilogrammes), added gradually, with or without decomposition of sulphite of soda; the stirring is continued until a sample of the mass dissolves in much cold water to yield an almost limpid solution, *i.e.*, only depositing after some time a slight flocculent precipitate, because it is found preferable not to push the conversion too far.

(*b*) 1,000 kilogrammes of 26° B. quebracho extract are similarly treated with 75 kilogrammes of caustic soda of 10° B. and 20 kilogrammes of sulphate of soda of 30° B.

(*c*) 1,000 kilogrammes of quebracho extract are heated in a rather large pan to 95° to 100° C., and 80 kilogrammes of soda crystals dissolved in 100 litres of hot water, added gradually with agitation, or an equivalent quantity of potash or bicarbonate of soda, or potash or ammonia, regulating the temperature and the addition of the alkaline solution, so that the mass does not prime; it is continued until a sample exhibits the solubility described in (*a*).

Summing up, we purpose to annex to our chief patent, and claim as our absolute and exclusive property:—

I. (*a*) The conversion of tannin extracts soluble with difficulty in cold water, such as quebracho, hemlock and pine extracts, into products more easily soluble, by means of appropriate hot treatment, by the hydroxides, sulphides or salts with an alkaline reaction of sodium, potassium or ammonium, in the presence or absence of bisulphites, sulphites or hydrosulphites.

(*b*) The preparation of soluble extracts, consisting in extracting quebracho wood or hemlock or European pine bark with water containing the products enumerated in (*a*).

(*c*) The processes described in (*a*) and (*b*) applied to mixtures containing other tannin matters, beyond those enumerated in (*a*) and (*b*).

II. The application of easily soluble extracts in tanning, alone or in admixture.

III. The products obtained according to I. (*a*), (*b*), (*c*).

Method of Application in Tanneries.—The solution of quebracho extract (only those soluble in the cold are meant) ought always to be carried out, as has been described, in regard to chestnut extract, *i.e.*, run the extract into the water, preferably at 35° to 40° C., or into the bark liquor intended for tanning.

Tanning of Sheep Skins.—It requires about 4 kilogrammes of quebracho tannin to tan twelve finished sheep skins of 8 kilogrammes. This quantity of skins is laid in a vat containing 50 to 60 litres (11 to 13·2 gallons) of weak bark liquor, to which there are added :—

TABLE XXX.—SHOWING THE PROGRESSIVE DAILY STRENGTHENING OF WEAK BARK LIQUOR IN THE TANNING OF SHEEP SKINS BY QUEBRACHO EXTRACT

First day, morning, first addition . . .	800	grammes	of tannin
„ „ two hours after, second addition .	800	„	„
Second day, morning, first addition . . .	800	„	„
„ „ two hours after, second addition .	800	„	„
Third day, one addition	800	„	„
	4,000	„	„

The skins ought to remain in contact with the bath liquor three or four days, then withdrawn, drained on the horse, washed in clean water, again drained, and finally laid out to dry screened from light. During the three first days of tanning the skins are agitated with a paddle mill; the last three days it suffices to stir the skins about three times throughout the day.

Tanning of Calf Leather.—To tan a calf skin of about 3 kilogrammes, it requires about 3 kilogrammes of quebracho dissolved in 25 litres of weak bark liquor. The skins are agitated in vats from five to six days, during which additions of extract are made, in the morning and afternoon, at the rate of 250 to 500 grammes, up to the completion of 3 kilogrammes. According to the nature of the skins and their thickness, the duration of the tanning may be prolonged a few days; it is the tanner himself who is the best judge in such a case. In any case, when the skins are tanned, they are drained on a "horse," washed in clean water, then drained and finally laid out to dry slowly.

Tanning of Heavy Leather.—Quebracho extract may also be used for tanning heavy leather, provided that it only be employed in the rehandlings, and in gradually increasing proportion, which may rise rather high.

Remarks.—In commencing tanning operations, it is indispensable to use weak and already used bark liquors. The baths, from which the tanned leather has been withdrawn, may serve as an initial bath for new leather before being put into a new bath; this is a method of utilising the tannin contained therein. It is evident that it is necessary to agitate the skins after each addition of extract, which ought to be previously diluted with bark liquor; on this work being done gradually and judiciously, depends the success of the tanning process.

CHAPTER VIII

MANUFACTURE AND USE OF SUMAC EXTRACT

Leaf Sumac.—The following varieties are found in commerce: *Sicilian Sumac* (*Rhus Coriaria*), which contains from 16 to 24 per cent. of tannin, is the most esteemed, and ought to be specified for the manufacture of extracts (the male sumac up to 27 per cent.); *Italian Sumac*, which titrates from 13 to 18 per cent.; *Spanish Sumac* (collected in the provinces of Malaga or the districts of Priego, of Malino, of Valladolid) is a somewhat esteemed variety, which contains 12 to 15 per cent. of tannin; *Tyrol Sumac*, very odoriferous; French or Donazère sumac, which titrates 13 to 14 per cent. of tannin; it is prepared in the valley of the Rhone, under the form of a dark green powder, the smell of which recalls that of the leather; *Redon Sumac*, which grows throughout the whole of the South of France; its tannin content equals that of the preceding; *American Sumac*, much used in admixture with Sicilian sumac by Morocco manufacturers and for the tanning of buff leather; it titrates 16 to 20 per cent. of tannin; *Virginian Sumac*, whose tannin content is variable. All these varieties contain a different tannic acid, but according to Stenhouse, the tannic acid of Sicilian sumac is identical with gallo-tannic acid. In damaged sumacs or those which have been warehoused too long in badly ventilated stores, the greater part of the tannic acid is transformed into gallic acid and glucose, with a deepening of the colour of the solutions of this old sumac.

Lentiscus.—One of the principal sophistications of Sicilian sumac is the addition of 20 to 60 per cent. of Lentiscus in powder, from the leaves or petioles of *Pistacia lentiscus*, a shrub which grows more especially in Tunis, in Algeria and in Corsica. This tannin principle barely contains more than 10 to 15 per cent. of tannin and does not give on leather the results furnished by Sicilian sumac. Tanners therefore would do well to make sure of the different qualities which are offered to them at prices which would indicate the extra-ventilated powdered quality (No. 1) or in leaf, but which in reality is a mixture "according to the formula" of Sumac Lentiscus-Tamarix.

Tamarix.—The leaves of this shrub, which grows in Tunis, in

Algeria and on the Mediterranean littoral, contain from 8 to 10 per cent. of tannin and are used to sophisticate Sicilian sumac.

Manufacture of Sumac Extracts.—It may be stated at the outset that the plant and equipment of a sumac extract factory are similar to those used for the manufacture of oak extract, *i.e.*, with a battery of open vats and triple effect plant for the evaporation of the sumac liquors. There are two kinds of sumac extracts, cold extracted and hot extracted. The first called "blonde liquor" or purified 30° extract is the product of sumac leaves macerated in the cold or at about 30° C.; the second is the product of the hot macerated leaves. Moreover, there are found in commerce other inferior qualities from the maceration of a mixture of sumac lentiscus or tamarix, or of liquors concentrated by a second hot maceration of leaves, already cold macerated.

Remarks on the Manufacture of Sumac Extracts.—The oxygen of the air, aided by atmospheric ammonia, produces on certain vegetable liquors the following remarkable effects: (1) Conversion of white indigo into blue indigo; (2) colourless buckthorn extract changed into Lokao or Chinese green; (3) orcine, the colourless principle of *Archil lichens*, passes to the condition of orcine, a violet colouring principle which for seventy years has rendered great services in the dyeing of wool and silk. So we can justly ascribe to oxygen the same action on 5° to 6° B. extracts extracted from sumac in the open air. In fact, the first wash water cold drawn from the Sicilian sumac is colourless, the second is less so, and the degree of coloration is accentuated more and more up to the seventh washing, owing to the aeration produced by the play or emulsion of the pump used to pass the liquors, which are pumped from below upwards and from one extractor to another, the latter seven times in succession. In order to ascertain the point at which this oxidation stops short, the authors spread 5° B. sumac extract in thin layers in the open air. After eight days it was completely black, passing through the following tints: yellow, rose, violet, blue and green, indices of the formation of a large quantity of gallic acid. Ether (extracted) tannin, of prime purity, brought likewise to 5° B. and exposed to the air in the same conditions, gave the same coloration result, but less rapidly, owing to the absence of the saccharine principles contained in sumac. It must therefore be concluded that tannins in a general way cannot support the action of the air without being influenced thereby, when they are diluted with a certain quantity of water, and that it is, therefore, of the greatest importance to prevent this contact during their extraction and their evaporation up to 25° and 30°, and even to the dry state. By pushing the evaporation to the dry condition they become unalterable and can then be

used as raw material for the manufacture of alcohol and ether tannins. For fine colours the loading of silk by tannins manufactured without excess of air would perhaps give the ideal of the colourless. These considerations apply not only to the manufacture of sumac extracts but also to all the tannin extracts which require, in virtue of their composition itself, to be protected from the oxidation of the air as soon as they are in the condition of weak liquor. There is quite a series of industrial experiments which should interest the chemists of this industry, which is capable of numerous improvements.

Cold Extract.—Leaf sumac, according to the *modus operandi* of the authors, undergoes seven successive washings with cold water, after two hours' contact in the case of each. The following are the respective strengths: 1st liquor, 6° B.; 2nd, 4° B.; 3rd, 3° B.; 4th, 2·2° B.; 5th, 1·2° B.; 6th, 0·6° to 0·7° B.; 7th, 0·1° to 0·2° B.

As the macerated leaves have to be fed into the gazogene (gas generator) furnaces in admixture with other waste, and as they absorb 275 per cent. of moisture, they are pressed in a mechanical press (100 to 150 kilogrammes) to recover again 225 per cent. of the dried macerated leaf, as liquor of 0·8°, corresponding to 8 to 9 per cent. of 20° extract, or 3 to 4 per cent. of dry sumac extract (it requires about 270 kilogrammes of 20° extract to produce 100 kilogrammes of dry extract). This recovery enters eventually into the ordinary working profit.

Yield.—As mentioned above, the yield in extract is a function of the quality of the sumac leaves treated; the yields, therefore, obtained by the authors were somewhat variable.

Sicilian Sumac No. 1, Superior Quality.—Yield in dry extract per cent. kilogrammes of leaves treated, 43 per cent.; in 25° extract, 92 per cent.; 20° extract, 110 per cent.; extraction done in the cold with recovery of the liquor contained in the marcs.

Hot Extraction, 80° to 90° C., of 25 Tons of No. 1 Palermo Leaf Sumac.—Yield in 25° extract, 78 per cent.; yield in 20° extract, 98 per cent.; in this yield the amount of extract obtained by the compression of the leaves after the seventh wash water, and which was used in the manufacture of the dry extract, has not been taken into account.

Cold Extraction.—Weight of sumac in powder treated, 2,000 kilogrammes; yield in 30° extract of powder, 55 per cent.; yield in 20° extract per cent. of powder, 83 per cent.

Other Yields.—Hot extraction of No. 1 Sicilian extract, superior harvest. 123 kilogrammes of leaves yielded 120 kilogrammes of 25° extract; cold extraction of the same quality of sumac as the preceding; 162 kilogrammes of leaves yielded 120 kilogrammes of 25°

extract. By pressing the leaves macerated with seven wash waters, and utilising the liquors, 0·8 per cent. of dry extract was obtained.

Qualities of Sumac Extract.—Sumac extract for transportation is delivered commercially in the liquid state and at 30° B., there is, however, a certain quantity of dry sumac extract consumed. As manufacturers of repute of this class of extracts, the authors would quote the firm of Jean Rod, Geigy & Co., of Bâle, who have been engaged in the manufacture of sumac and logwood extracts since 1856, and whose brands are universally appreciated, such as purified 30° sumac extract with 31·8 per cent. of tannin; surfin 30° with 32·4 per cent.; O 30° with 32 per cent.; B 30° with 25·5 per cent.; BO 30° with 23·7 per cent. These extracts are sold, according to quality, from 40 to 62 francs. In France the authors would quote the firms of Watrigant et Fils, of Marquettes-lez-Lille; Dubosc Frères, of Havre; Huillard et Cie., of Suresnes.

Use in Tanning.—Sumac extract is specially used to bleach leathers to be dyed rather than to tan them, because it tans too soft; it is used especially for leathers intended for high-class boot uppers, binding and Morocco work. It is also adapted for loading white or pale-coloured silks.

Gallo-sumac.—Gallo-sumacs, made by the authors in large quantities, are mixtures in variable proportions of divi-divi, sumac and chestnut, which give good yields and extracts appreciated by tanners on account of their high tannin content, which exceeds 32 per cent.

CHAPTER IX

KHAKI SUBSTITUTE FOR QUEBRACHO—ITS USE IN TANNING

This tanning material, recently placed in the market, would appear to come from Borneo (*viâ* Singapore); it is extracted from a leaf and put on the market in a dry state. Khaki contains about 60 per cent. of tanning matter. It is a rapid tannin and an astringent one. It must, therefore, be used with moderation, preferably with other mild tannins, otherwise the pores of the leather may be contracted and its tanning retarded. It yields a firm but soft leather, and less hollow than quebracho. Nourishing the leather by its assimilative capacity, it renders it waterproof. Khaki gives more weight, a more thick substance and a heavier leather than gambier, and in that respect it is much superior to quebracho, mangrove, mimosa, and other extracts used alone. It does not contain any resinous body, like hemlock (spruce) and quebracho, which often are the cause of the spots or marblings to be seen on leather. It contains less than 1 per cent. of insoluble matter. In concentrated solution it does not separate as dry quebracho extract does. A strong liquor of khaki and hot water, or of khaki and a hot liquor consisting of other tanning materials, deposits naturally on cooling, but the deposit formed may be redissolved easily by taking it up with a certain quantity of hot water. This deposit is of a flocculent nature, having none of the resinous gluey properties so unpleasant in quebracho, mangroves, or the barks of mallet. Dry khaki extract is dissolved in the same way as chestnut (see above).

Method of Application in Tanning.—Khaki, being a tannin very soluble in water, may be employed in tanning, in admixture with other tannins, with which it gives good results, such as: Into a tan pit pump a myrobolam liquor, add 600 kilogrammes of gambier and 300 kilogrammes of khaki, dissolve by an open steam pipe at about 40° to 45° C., use this liquor afterwards either for the final tanning of leather or for the retanning of split leather. This formula is constantly used by English tanners, who obtain with this mixture first-class leathers. Another mixture consists of $\frac{1}{5}$ khaki, $\frac{1}{5}$ gambier, $\frac{3}{5}$ myrobolam and oak bark and drenching with the preceding strong liquor. Khaki has

also been used with success, by mixing it with chestnut extract in proportions varying from $\frac{1}{10}$ to $\frac{1}{6}$ of the tanning substances used, so as to mitigate the reddish colour of the khaki. Finally, some tanners have used the following mixture: Khaki $\frac{1}{2}$, chestnut extract $\frac{1}{2}$, to drench the semi-rapid tanning pits, and with liquors of 5° to 7° B. If the tanners use a system adopted in Germany, *i.e.*, swell the leathers first in the pit with an organic acid, then dye them in the dye pits, and then lay them out in dry layers, it is advisable to add in the case of the first layers, to each fifty skins, four to six buckets of this strong khaki to the liquors used for drenching the said pits.

Tanning of Sole Leather (lissé).—Khaki may be used according to the following general formula for the manufacture of sole leather, as is done in Germany and in Austria. The general system of tanning in Germany is to place the skins, after careful working in river water, to be dyed in a series of pits and lasting several weeks, then directly to the layer pits. These layers consist of bark and strong liquor, to which extracts are added to strengthen them. For this kind of tanning it is advisable in the first, the second and perhaps in the third steep to dissolve the khaki in the liquor itself of the pits: 50 kilogrammes of khaki per 100 skins. The other system more resembles British tanning because khaki is used in the "handling" or in the powdering vats, at the rate of 50 kilogrammes (say 1 cwt.) per pit and sixty hides dissolved in the myrobolam liquor used for drenching the skins.

Finally, certain tanners use it with success along with oak bark, valonia, myrobolam, chestnut extract, in proportions which vary with the requirements and the exigencies of their tanning which practice alone can guide.

CHAPTER X

VARIOUS TANNING SUBSTANCES—THEIR MANUFACTURE AND USES

1. *Divi-Divi*—2. *Valonia*—3. *Chinese Galls*—4. *Myrobolam*—5. *Palmetto*—6. *Mimosa*—7. *Tara*—8. *Mangrove* (*Palétuvier*)—*R. Catechu or R. Gambier*

1. *Divi-Divi.*—This tanning substance is found on the market in the form of pods or siliquas of about 5 to 6 cm. long, fleshy, reddish yellow, bent in the form of an S, and containing hard, smooth, ovoid seeds; they are produced by the *Cæsalpinia coriaria,* which grows in South America and which also yields a bark used in tanning. The pods or siliquas contain about 30 per cent. of tannin, whilst the bark only contains 12 to 15 per cent.

Manufacture of Divi-Divi Extract.—The divi-divi pods are exhausted by seven washings of two hours each at 90° C. (194° F.); then the first washing and the half of the second are evaporated *in vacuo* up to 30° B. The following are the strength in degrees Beaumé of the different liquors: First, 8·5°; second, 4·3°; third, 3°; fourth, 1·9°; fifth, 1·3°; sixth, 0·7°; seventh, 0·3°.

Yield from Lot of 50,000 Kilogrammes Treated (*Maracabe and Rio-Aiche Quality*).—100 kilogrammes of pods yielded 117 kilogrammes of 30° extract; 85 kilogrammes of pods yielded 100 kilogrammes of 30° extract; 168 kilogrammes of pods yielded 100 kilogrammes of dry extract. Divi-divi extract of 30° generally contains 30 to 33 per cent. of tannin, the dry extract 53 to 55 per cent. The extract, itself at 30°, being essentially fermentable, it is important to asepticise it by a small addition of formic acid or mercuric iodide. The priming of the vats by the too intense fermentation developed in summer is thus avoided. By its great yield in extract it will be seen that this tanning principle is interesting to the tanner who uses it, especially in tanning light pliant leather, decolorised extracts yielding very bright tints.

2. *Valonia.*—Valonias are the scaly cupules of the *Quercus Œgilops* (Turkey Oak), a tree common to the Greek Archipelago. German, Austrian, Italian and especially British tanneries consume large quantities of it. Valonia is mixed with oak bark for the tanning of sole leather in the two last powders; the resultant leather is more water-

proof than that tanned by oak bark alone. It is in fact one of the best tanning agents at present on the market, and British tanners make excellent use of it, if one can judge from the leathers imported into our (French) market. The chief market is Smyrna, which exports all the known varieties: Caramania 1*a* and 2*a*, Camatina, bons refusos, English 2*a* without trynacks, uso-English, unacqua, trynacks trillos 2*a* or 1*a* sifted, Greek dragoman; the price of these different qualities varies from 16 to 27 francs c.i.f. Havre, Dunkirk, Antwerp. As the tannin content of valonias may vary from 15 to 35 per cent., according to quality, source, etc., it is very important when buying to determine their tannin content, the more so as the percentage of extractive matter is also very variable.

Manufacture of Valonia Extract.—The authors had occasion to treat 10 tons of a lot of mixed valonias which received seven wash waters at 95° C. (203° F.), each of one and a half hours. Strength of the respective liquors: (1) 5·3° B.; (2) 3° B.; (3) 1·2° B.; (4) 0·8° B.; (5) 0·5° B.; (6) 0·2° B.; (7) 0·1° B. The average strength of the liquors was 4°; after cooling, decolorisation and evaporation *in vacuo*, they yielded a 25° extract, the tannin content of which was 26 per cent. Yield: 117 kilogrammes of cupules produced 100 kilogrammes of 25° extract, say 85 per cent. of the weight of the valonias treated.

3. *Chinese Galls.*—This tanning agent, the product of a species of sumac, contains 50 to 60 per cent. of tannin; this high tannin content ought to draw the attention of tanners to its commerce and its production in Eastern Asia. Treated in the same way as divi-divi or valonia, its yield is 125 to 128 in dry 30° extract, *i.e.*, 78 kilogrammes of Chinese galls yield 100 kilogrammes of 30° extract.

4. *Myrobolam or Myrobolans.*—This tanning agent comes to us from India as rounded, greyish-black fruits, collected from several species of large trees (Terminalia) and containing, according to source and year, 25 to 35 per cent. of tannin. The authors had occasion to treat some tons of Jubelpoore I.-II. by seven washings at 90° to 95° C. (= 194° to 203° F.). The following are the results obtained: Yield in 20° extract per cent. of myrobolam treated = 76 per cent., of 25° extract = 60 per cent. Weight of myrobolam to produce 100 kilogrammes of 20° extract = 145 kilogrammes. The liquor obtained, which titrated 37°, decants easily and rapidly; evaporated at 65 mm. (? cm.) of vacuum, it yielded a very fluid extract of a yellow colour, used in tanning goat skins a brilliant yellow and endowed with great pliancy. Moreover, it is a tannin of universal repute which mixes very well with bark liquor or chestnut extract. The chief makers of French and foreign myrobolam extract are Compagnie Française des Extraits Tannantes et Colorantes, Watrigant et Fils, etc.

5. *Palmetto Extract.*—This tanning agent is extracted from the roots of the *Sabal surrulata,* which grows on the sandy maritime soils of Florida, and manufactured by the Florida Extract Company, Titusville, Florida. The chief use of palmetto extract is as a gambier substitute. The following are the properties which give palmetto an advantage over gambier: "It tans more rapidly, gives a better weight, produces stronger leather by causing it to swell, a leather which may undergo a higher temperature in varnishing". Palmetto extract, this new tanning agent, seems to be destined to play a very important part in the tanning world. Precise data as well as instructions on the method of applying it to the most different kinds of leather now follow, based on the numerous experiments and laborious researches of Professor Kohnstein of the leather factory of Pfistervogel, Milwaukee. Palmetto extract is sold in American tanneries by the waggon-load, and its consumption increases in proportion as it is adopted in actual practice and that the practitioner recognises its valuable qualities. In the beginning the high figures in the percentage of non-tannins, determined in the laboratory, could not be sanctioned, and moreover also all sorts of difficulties combined to throw discredit on the use of palmetto extract. First of all, it was the difficult solubility of the extract in cold water, and also the fact that the extract seemed to become darker when exposed to the air, but especially to light. In spite of these unfavourable observations, demonstrated in the laboratory and in the workshop of tanneries, Kohnstein did not abandon his researches but continued his experiments with this new product. The experience acquired in the manufacture of the extract and in tanning showed that the same difficulties had to be overcome as in the production of tanning extract from the pine and oak wood and quebracho extract. In fact, with the best bark and the finest calf skin, very bad leather may be made if the operator has no knowledge of tanning processes. To tan by means of palmetto extract, it requires as much experience as in working with the best oak bark. Thanks to laboratory researches and to the experience acquired in the factories themselves, it is now possible to supply the chemist as well as the practitioner with some valuable advice concerning the application of the extract and its judicious use. The preliminary treatment of the leather must also be explained, because it is very important in tanning with palmetto extract. This treatment depends on the characteristic properties of leather. The Florida Extract Company have lately sent out samples of extract of the following average chemical composition: Density, 30° B.; water, 52·94; ash, 11·09; tannin, 25·96; organic substances incapable of being absorbed by the skin, 10·31 per cent. The extract being

soluble in cold water at a temperature of 65° F., yields a bright red colour.

In analysing palmetto extract it has been remarked that the skin powder is dissolved to a certain extent by the inorganic substances of the extract, which are partially of an alkaline nature. Attention has first of all to be drawn to a point already demonstrated in previous publications, *viz.*, that palmetto extract contains natural alkaline chlorides and bromides. See the laboratory researches of the State of Florida of 17th June, 1898, in which are published the analyses of palmetto root by the State chemist, W. A. Rawls, from which it will be seen why chemists found so much non-tannins, and that because the skin powder dissolved is also weighed and brought into the calculation at the expense of the actual percentage of tannin. This error was pointed out at the time when, in 1897, the Congress of Leather Chemists sat in London. The same error occurs with extract to which borax is added, either as an antiseptic or to accelerate the solubility in cold water. If a certain quantity of palmetto extract be ignited, and the residual ash dissolved in water and filtered through skin powder, working on weighed quantities, the chemist will be able to acknowledge the correctness of these remarks, and he can then afterwards avoid the said error. Palmetto extract is chiefly intended as a substitute for gambier or for use in admixture therewith. The following are in a few words its advantages: "Palmetto extract gives a better weight, tans more rapidly and renders the leather thicker by causing it to swell, finally it stands heat better during greasing in the cask, during varnishing and the drying of the varnish in the stove (that is, during japanning)". These valuable properties have led to palmetto extract being used for the most different kinds of leather. The dark colour of the leather may be explained and the inconvenience incidental thereto be remedied, chiefly by drying, when it has been exposed for some time to broad daylight. The practical tanner knows that tannin solutions containing alkaline salts easily absorb oxygen and become darker, but if the skins be treated with a little soap or grease the alkali is neutralised by the grease, and the good effect of a fat liquor is obtained on the leather, producing besides excellent quality a very fine colour. In retanning chrome leather, such salts are very useful in so far as they neutralise the traces of acid remaining in the leather, and by forming, in so far as they combine with the tannin of palmetto, a superb mordant for leathers to be dyed black or by aniline dyes. Silk, woollen and cotton dyers have not been slow to appreciate in their turn the value of this mordant, seeing that palmetto extract is free from resin and fat, and that it penetrates easily into the fibre. In retanning chrome leather with pal-

metto extract excellent results are obtained, which it is in vain to seek for with other tannins; the grain remains soft, does not become rough nor slack, and produces a very soft texture of excellent appearance.

Tanning of Calf Skins and Kips.—The crude green salted skins are first passed into fresh water, then into a bath, twice renewed, then the third day they are put through the paring machine, and steeped in water during the night. From the water the skins are put into the lime vats. The first lime is left till it gets "killed," and the skins are left in it a day and covered each day with a better layer of lime until they pass the fifth day in a fresh lime vat. In the case of fresh skins there are taken for every 3,000 lb. of raw skins 150 lb. of lime and 8 lb. of arsenic. To strengthen the succeeding limes there must be taken, each time the skins are turned, 70 lb. of fresh lime and 4 lb. of arsenic. The sixth day the skins are unhaired and put into milk of lime for twenty-four hours. For 3,000 lb. of raw skins there are taken for this solution 70 lb. of pure lime dissolved in water. After that operation the skins are repared, the heads are split, washed and finally assorted.

For a chrome mixture the skins require a bran mordant; for tanning by the "Palmetto Extract Method," or for mixtures with other vegetable tanning agents, a mordant of pigeons' dung is recommended. For "glazed kid," goat, lamb and sheep skins, intended to be dyed, so as to prepare them for tanning by palmetto extract and chrome, dogs' dung is to be used. After paring the skins and smoothing the grain they are ready for tanning. They are then steeped in a palmetto liquor of 8° Barkometer and removed in half an hour. They are then tanned in a fulling machine by means of palmetto extract of 30° B. (51° Twaddle), heated up to 35° C. (95 F.). For 700 lb., the weight being taken after the skins leave the beam-house, 500 lb. (one barrel) of extract is run into the vat. So as to prevent the skins from twisting and getting entangled they must be inspected from time to time, and the fulling machines adjusted in such a way that the paddles make the same number of rotations in both directions. After having been continuously mixed for five or six hours the skin is well tanned, even in the strongest places of the nape of the neck and the cheeks, without drawing or roughening the grain. The skins are then rinsed in a light liquor, in which the skins are dyed and pressed. They should then be passed into a weak palmetto liquor of 8° Barkometer, and retanned one hour in a strong solution, in the fulling machine. When taken out of the fulling machine they are washed in lukewarm water. This liquor is used as the dyeing liquor. When the skins have been washed and pressed, they are thrown directly into a fulling machine

containing heavy liquor. The fulling machine is heated by steam to a temperature of 140° F. For 440 lb. of pressed leather 5 lb. of soap, 4 litres (say 1 gallon) of sod oil heated in half a cask filled with water to a temperature of 120° F., are used, treating the mass in the fulling machine for half an hour. The door is then opened and water run on to the skins to wash them, after which they are hung up to dry. The grain of the leather may easily be protected if the skins are to be dyed. So if the dry skins have accumulated a few days, they may be joined to the different kinds of leather, and be, after a preliminary treatment with Sicilian sumac, as good for varnished and dyed leather as gambier leather.

Tanning of Cow-hide Sole Leather, Harness Leather for Saddle-girths, Machine Bands, etc.—The well-drenched and washed skins are scraped and dipped into lime vats strongly drenched with sodium sulphide. For 3,000 lb. of raw hides, 210 lb. of lime and 20 lb. of sodium sulphide are used, and 75 lb. of lime and 10 lb. of sodium sulphide are used to strengthen the reagents. After having been coated with lime for five days, the skins are unhaired by the unhairing machine, again unhaired, then polished by hand and rescraped; then after being washed with cold water they are mordanted with cold muriatic acid—18 lb. acid per 3,000 lb. of hides; then they are treated with a bath of pigeons' dung for two hours, with constant agitation. The skins are then scraped and hung in vats filled with dye of 12° density, which may be brought to 20°. With the first palmetto bath it is advisable to leave spaces between the skins by suspending them, so as to facilitate uniformity in the dyeing of the grain. The sixth day the hides are lifted with the poles, and laid flat in palmetto extract of 30° B., turning them twice the first day. So that no loss nor leakage of extract may occur, it is advisable to place the hides on oblique boards. The skins are tanned in four days: it will then be seen that the extract, without any motion, has fermented even the densest parts on the top. The hides are slightly rinsed in weak palmetto extract, exposed to the air and fleshed. After fleshing, the hides are re-tanned with weak palmetto liquor, and finally fulled in a fulling vat of 30° B. for six hours. To obtain a bright yellow effect with this kind of leather on drying, it is advisable to use the fat liquor called "sod oil" (*Moellon degras*) described above. Moreover, palmetto extract is well adapted for tanning split leather, for the production of heavy leather for soles, in combination with other tannins, such as valonia and oak. Finally, combined with gambier it is an excellent medium for softening leather, and imparting to it a greater capacity to resist heat.

Chrome Tanning with Palmetto.—No other tannin agent is so well adapted for use in conjunction with chrome as palmetto extract. The Florida Extract Company of Titusville were the first to discover this peculiarity, and it is to them that palmetto extract on chrome owes its reputation for woollen and cotton dyeing. For hides tanned in two chrome baths, it is advisable to retan in a palmetto extract liquor. For 300 lb. of pressed chrome hides 23 lb. of palmetto extract, with 1 pint of glycerine, dissolved in a cask of water are used. Chromed leather treated in this way is exceedingly well adapted for being dyed, the grain becomes firmer, and is not detached on rubbing. Aniline dyes, logwood, or other dyewoods, such as fustic or redwood, are more fast, do not become dirty, and are easily glazed. This leather is also fit for taking sod oil (*Moellon degras*); it preserves its silky grain—that is why it is the best material to use for dyed chrome leather for gloves. Hides which are to be tanned after the chrome bath, may be treated similarly; however, the chrome bath may follow the tanning with palmetto. Above all, palmetto extract is distinguished by the fact, that with 28 per cent. of tannin it is very liquid, penetrates the main body of the leather very energetically, in spite of the concentration of 30° B., and is consequently much fitter for tanning by Durio's process.

Another Method of Tanning by Palmetto Extract. Tanning of Goat, Sheep and Calf Skins with that Extract according to the "Shoe and Leather Reporter".—Lately, and especially in America, tanning with palmetto extract has assumed enormous developments, because it yields a very good soft and full upper leather. Recently it has also been used for goat, sheep and calf skins, and the process is as follows. First the skins are drenched with special care and attention. It is especially necessary to use arsenic depilatories, and it is important that the skins are laid perfectly soft and in good order in the depilatory. These are preferable to the ordinary depilatory, because a better grain and a firmer texture are imparted to the skin, and these are precisely the qualities required in goat and sheep-skin leather. An old depilatory is used in the beginning, in which the skins are laid for some days, then they are passed through depilatories of gradually increasing strength, one after the other, until the unhairing is complete. It is not believed to be desirable to use lime liquors in the beginning, but the skins may be placed, after five or six days, in quite fresh lime, after passing through several depilatories. For the preparation of the last, for 1 ton of skins 1 cwt. of lime and 6½ lb. of arsenic are used. The third day the skins are taken out and unhaired, and if a very fine skin be desired they are replaced in fresh lime for twenty-four hours, after which the skins are scraped and are ready for tanning.

Branning.—The skins are placed in a bran steep, and good results are obtained as follows. Half a ton of bran is taken for 400 heavy or 450 light skins, and run into a cask filled with enough water, which forms a greasy paste which is soured, which takes about forty-eight hours. There is added to this "branning" medium about 1 litre to $1\frac{1}{4}$ litres of sulphuric acid, and 93 to 95 kilogrammes (204·6 – 210 lb.) of pure salt, so as to get a density of 58°. The skins are stirred in this liquor for three to five hours, depending on the depilatory and the amount of lime which they contain; they are then withdrawn. This maceration renders the skins pliant, and gives them a fine grain. The skins are prepared for tanning by gentle washing in lukewarm water, working them softly on the grain.

Tanning.—To feed them to the end palmetto extract is used. The skins are first weighed, if they come from the previous workshop, in good working order. They are then laid in a weak bark liquor of 8° Barkometer, in which they are stirred for about thirty minutes. They are then put with constant stirring into a palmetto liquor of 35° Barkometer heated to about 47° C. (122·6° F.). A liquor is prepared for 100 skins, about 65 kilogrammes (143 lb.) of palmetto extract and the necessary water. Tanning is complete in five to six hours. It is not necessary that light skins be retanned, but it is better to tan heavy skins once more after cleaning and stretching, and in that case, fulling the skins a second time about an hour in a weak liquor. When tanning is complete the skins are taken out of the vat and washed perfectly in lukewarm water; they are pressed and treated with sod oil (fat liquor.)

Finishing.—The leather may be finished in two different ways: it may be passed into the "fat liquor" (sod oil), dried, coloured, finished, or it may be oiled in the ordinary way in the fulling mill, then dried, again oiled, dyed and finished. The first method is better than the second, as the leather is better looking, and labour is saved. The method of preparing the "fat liquor" (sod oil) is described farther on. The skins are well rubbed with this lubricant and fulled for half an hour in the fulling machine; they are then laid out to dry. The dried skins are drenched with lukewarm water and coloured blue on the flesh side with crystallised logwood extract and flesh stain D, and on the grain side with leather black; they are oiled both sides, again dried, stretched, and finished in the ordinary way. The different sorts of Levant Ink (inks to be used before finishing) may also be used (according to whether black or pale leather is to be made).

Feeding for Use in the Churn. Fat Liquor P.—For 100 kilogrammes of skins take $2\frac{1}{2}$ to 4 kilogrammes of this composition. Soft skins require the minimum, hard skins the maximum quantity. Weigh the desired

quantity of fat liquor and place it in the churn with half the water to be used. Do not boil, but heat to 40° Re., and stir until solution is complete. Then add the other half of the water to bring the liquid to the proper temperature for feeding. Then proceed as usual.

Oil Emulsion to be Rubbed into the Skins. Fat Liquor A.—Take 2 kilogrammes of this emulsion, heat it to boiling, add 1 litre of water; then rub this composition into the skin with a woollen rag, at a temperature of about 60° C. (140° F.). When the skins are treated, instead of placing them flower upon flower, they must be placed flesh upon flower. If the emulsion be heated by a steam jet, it will not be necessary to add the above litre of water, because the condensed steam will suffice. The skins when treated are taken to be dried, then when dried they are left in a heap for four to seven days, then moistened and passed through the opening machine and finally placed on the *estrèque*. They are then ready for drenching before finishing.

6. *Mimosa.*—These (wattle) barks, which come from Australia, are the product of different varieties of acacia, such as pyenantha, cyanophylla, leiophylla, mollissima, etc. They have been cultivated for some years both in Tunis and Algeria with good results and even in the South of France, where their culture might be developed with success, the mimosa being a hardy shrub which requires but little care and is very productive, as certain mimosas shoot continuously from the stump after being felled, and a tree barked when six years old may yield 20 lb. of bark. Moreover, a hectare of mimosas may yield according to the quality and nature of the tree and the climate in which it grows, 4 to 5 tons of bark per hectare (say 32 to 40 cwt. per acre). The mimosa bark of Natal, one of the most justly appreciated, contains 34 to 36 per cent. of tannin, and may yield 110 to 112 kilogrammes of 25 per cent. bark, say 90 kilogrammes of bark to produce 100 kilogrammes of 25° mimosa extract. With mixed qualities the authors obtained 103 per cent. in one case, and 96 per cent. of 25° extract in the treatment of bark of Algerian origin. This bark yields on maceration liquors which easily titrate 5° to 6° B., which, when concentrated in a vacuum of 65 c.cm., yield an extract readily soluble in cold water without any decolorisation or clarification. It is thus an excellent tanning material for the manufacturing of tannin extracts, the more so as it yields in tanning fine coloured leather of superior quality. The erection of mimosa extract factories in Australia and even in Algeria will give rise to a series of interesting commercial transactions for some time to come. The following are the yields from Hyères (Var) mimosa bark treated like the foregoing: Yield per cent. of bark in 20° extract = 80 per cent.; in 25° extract = 64 per cent. Quantity of bark required to produce 100 kilo-

grammes 25° = 156 kilogrammes. The authors found this bark to yield on analysis 29 per cent. of tannin and the corresponding 25° extract 32 per cent. of tanning matter. These yields may be taken as commercial, as the experiments were made on half a ton of bark. It will thus be seen how desirable it is to form plantations of, and cultivate, this genus, looking to the yield of bark per hectare of mimosa and that of the extract per 100 kilogrammes treated. As a working basis, 2,000 to 3,000 hectares (say 5,000 to 7,500 acres) planted in mimosa would suffice to supply a factory treating 25 tons which would produce 15 tons of 25° extract. Extractors will soon be compelled by force of circumstances to use tropical tanning material (they have already made a partial start), either by treating them on the spot, or by acclimatising them in the (French) colonies or on French soil, for capital is best utilised in the development of prosperous industries, and it may be justly said that the tannin industry is one of the most lucrative. The price of mimosa bark oscillates according to its source between 18 and 27 francs per 100 kilogrammes for good qualities c.i.f. Havre or Marseilles (say 7s. to 10s. 6d. per cwt.).

7. *Tara.*—Tara is a tanning material which comes from Chili and Peru, in the form of pods, like divi-divi but paler. In Peru it is used for tanning seal skins, yielding a colour like that obtained from evergreen oak alone. It contains 30 to 35 per cent. of tanning material and a large proportion of gummy principles, which render it difficult of being accepted by extract makers, unless it be got rid of solely in admixture. It is sold in French European ports at 30 francs the 100 kilogrammes (say 12s. the cwt.).

Method of Using Tara.—Take 100 butchery hides, weighing 30 kilogrammes (66 lb.) in bulk, say a total of 3 metric tons, or the equivalent of this weight in cut hides, sheep skins or goat skins or any other kind of skins. To tan that quantity will require 600 to 700 kilogrammes (1,320 to 1,540 lb.) of tara. Tanning is complete within four days of laying in the pit. All skins to be tanned with tara ought to be first passed through the preliminary baths, for preparing the skins, to bring them into the desired condition for being laid in the pit. The first day the skins are placed in a pit large enough to allow one or two men to manipulate them with ease. The skins are laid in the pit, folded in two, in the direction of their length, the flesh side on the outside. The tara is sprinkled on that side, placing the skins as they are usually superimposed. It is essential that, on the first day, only 100 kilogrammes (say 2 cwt.) of tara be used (for it contains gummy principles which harden the leather if used immediately in too large amount). When the skins have been laid in the pit, they are first drenched with water previously saturated with

100 kilogrammes (2 cwt.) of tara and in sufficient amount for the skins to be completely steeped in it. The second day the skins are moved about by the workmen. This done, they are taken out of the pit, taking care to shake them well, so as to cause any adherent tara to drop off, and in proportion as it does so, it is collected and run into a vat filled with water in which 50 kilogrammes (1 cwt.) of tara have been laid the previous day, so as to be well saturated therewith. Then, the pit being emptied and well cleaned, the skins are laid into it again as on the first day, spreading a layer of tara between each skin, always on the flesh side, but taking care not to place the second layer on the part which has received the first. Then the skins are drenched with water from the vat above mentioned. For the second layer, 150 kilogrammes (3 cwt.) of tara are used. The operations of the first day are repeated. On the fourth day the skins are taken out of the pit, and after being drained are placed in a bath of fresh water previously saturated with 12 kilogrammes (26·4 lb.) of alum and 6 kilogrammes (13·2 lb.) of salt, and that for 100 skins. The quantity of water used ought to be just sufficient to cover the skins; they are then worked about and withdrawn from the vat, either to be dried or curried.

8. *Mangrove.*—The tanning agent is so designated in English and in German (in French *palétuvier*). It is also termed "mangle," a derivation showing its origin from the *Rhizophora mangle* growing in the Gaboon, Senegal, South America, India, Madagascar. It is being marketed in ever-increasing quantities as a reddish, more or less pale bark, hard, with a section showing black or white spots. According to quality and origin, it titrates 20 to 30 per cent. of tannin. Several foreign manufacturers have already used it in admixture in the making of quebracho extract, in which it retains its red colour and increases the tannin content of the quebracho. From experiments made by the authors to determine the value of this bark, they obtained a yield which oscillated between 80 and 90 per cent. of the bark treated, showing that it is a product of interest to extract manufacturers. As to its value for tanning, it is but middling; used alone, it can only yield a soft tanned leather, but it can be used in a mixture of the following composition, which has been already utilised: 40 parts pine bark, 20 parts oak bark, 30 parts mangrove, 10 parts mimosa. Good results are also obtained by mixing mangrove extract, now made in a way with which no fault can be found, with myrobolam, mimosa, oak, chestnut, according to the quality and nature of the skins to be tanned.

R. Catechu. Special Extract for Dyeing and Tanning.—"I have received a product which perfectly answers my requirements. It not only replaces japonica and other catechus, but it is also a really ideal

tanning material (*geradezu ideal*). The manufacturers, Paul Gulden & Co., call their new product R. Catechu" (W. Eitner).

R. Catechu. Special Extract.—R. Catechu is a product which can perfectly replace not only the various cutches in tanning and dyeing, but also gambier and japonica, because it possesses the different properties of these materials whilst greatly surpassing them in strength. Besides it yields better and brighter liquors, and runs much cheaper. R. Catechu does not fluctuate greatly in price like gambier and other tannins. As R. Catechu can be used in equal proportion with other tannins, it can not only be used for all the purposes for which they are used in tanning, but it may be used preferably instead of gambier, etc., because it renders the finishing of certain leathers more easy, in modern tanning. The proportions for the use of R. Catechu, by a competent man, for all tanning purposes are now given. Its use has been very advantageous, and principally in the preparation of new kinds of leather.

Dyed Calf (*American Method*).—The skins in bands (*en tripe*) pass through a series of dyes, which is made like that with japonica and which consist, according to the weight of the skin, of six to eight dyes; the last dye is for heavy skins, for which eight dyes of 24° Barkometer are necessary; for light skins six dyes of 24° Barkometer suffice. The striking colour is itself applied at 7° to 8°. After this tanning the leather is finished as usual. For bright colours the skin is repassed.

Dyed Calf (*Mixed Method*).—R. Catechu is more advantageous in mixed than in ordinary tanning, and in this respect surpasses all other tanning substances. The skins in prepared bands are treated for an hour in a solution of 5 per cent. on the naked weight of salt and 2 per cent. of alum, both dissolved in fifty times their weight of water, then beaten for twelve hours on a block and left to stand. Tanning is done as above in six colours, the first of which need only be of 16° Barkometer. The skin remains two days in each dye, a solution of R. Catechu is added each day to bring the strength up to normal. To increase the strength of the tanning liquor R. Catechu dissolved in twice its weight of water is used. The complementary finishing is done as usual.

Final Treatment of Chrome Calf with R. Catechu.—Chrome-tanned leathers which have been steeped once are treated, after neutralisation and folding, with a liquor composed as follows: For 100 kilogrammes (220 lb.) of folded weight, 3 kilogrammes (6·6 lb.) of soft soap dissolved in 50 litres (11 gallons) of water, to which 600 grammes (1·32 lb.) of potash are added, then 3 kilogrammes (6·6 lb.) liquid R. Catechu, or a solution of 1·8 kilogrammes (4 lb.) of solid R. Catechu, finally 3 kilo-

grammes of bone fat and ½ kilogramme (1¼ lb.) of vaseline. The skins are treated with this liquor for forty minutes to one hour in the fulling mill, they are then beaten on the block and left at rest for six hours so that they may become pliant. The skins are then stretched and dried or their working is continued. The dried leather ready for the tannery may be afterwards finished like black leather for dyeing. The latter receives in the cask, before dyeing, the final treatment with R. Catechu; in that case for 100 lb. of folded weight 3 lb. of liquid R. Catechu are used.

Mixed Chrome Tanning (*Dongola Chrome*).—The skins in bands, prepared for chrome tanning, are tanned in a simple chrome bath, *i.e.*, with one dip. The tanning is started with a previously used chrome liquor of 12° B., adding according to the thickness of the skin gradually, in six to ten hours, 25 per cent. of the weight of the naked extract of liquid chrome (Cromal). After tanning stand twelve hours on the block, then neutralise with 2 per cent. of sodium sulphide, wash, stretch and fold. Then follows the second tanning with R. Catechu as a colour basis (*Haspel farbe*). The finishing colour (*Eintreib farbe*) 5°, other colours 6° Barkometer, which are kept at 6° by adding the necessary amount to strengthen the liquor. The tanning process lasts, according to the thickness of the skins, from four to twelve days. Leathers treated in this way may be prepared like "leather by measure" for current articles, that is like leathers ready for bark tanning; if leather by weight be desired they are prepared like the latter. In dyeing the glaze of the varnish, for the preparation of nappa leathers, japonica or gambier was formerly used. R. Catechu produces a surprising effect, because it does not tarnish the colour, whilst it yields a pliant and elastic and at the same time a strong leather. Glacé skins are re-washed in two waters and then receive the dye bath prepared with R. Catechu and a vegetable colour; they are then steeped and afterwards passed on to be treated with the egg-yolk mixture and the final dyeing on the table. For an average skin 30 grammes (rather over an ounce) of R. Catechu are used. In dyeing with aniline dyes the leather is treated after washing only with R. Catechu. Then it is dyed with an aniline dye in the fulling mill, the colour is afterwards fixed, the leather rinsed and finally treated with egg-yolk mixture.

R. Catechu. A New Tanning Agent for Mixed Tanning.[1]—We recall here the old obsolete tanning experiments now forgotten, as well as new applications in the working of mixed tanning. One of these is fat emulsion, which may presently become of great importance. In mixed tanning two methods are used for preparing the leather, *i.e.*,

[1] According to Eitner.

mineral and vegetable tanning, which are distinguished by several properties in the different processes for preparing mixed leather, and which will go on increasing later on, according to custom, to a greater or less extent. As an example of mixed leather prepared almost by the old process, we may quote that done on the same principle as the Danish method for gloves and bandages prepared with alum and the vegetable tanning which follows afterwards, which is distinguished from alum leather by its softness and its strength. Eitner found in the stuffing of an armour a mixed leather of this nature, dating, according to the archives, from the time of Marguerite Maultasch, from which it may be deduced that mixed tanning was known long ago, and that it was known how to appreciate it at that epoch. The old process of mixed tanning can now no longer be taken as the starting-point of that of to-day, as mentioned above, because here, moreover, it is the outcome of chrome tanning. As already pointed out by Eitner in his work on chrome tanning, the imperfections of this method of tanning have been attributed to this fact, *viz.*, that with this process leather cannot be obtained with its full yield and that its preparation causes difficulties; finally, a badly tanned product is obtained, the result of the chrome tan on the vegetable tan. Vegetable tanning is carried out to-day on almost all skins already treated by chrome, and on a larger scale on the leathers sold as mixed leather. Thus, with mixed leathers treated by alum, those may be distinguished which have been more or less treated with vegetable tan. This replacing of mineral tanning by vegetable tanning is not easily accomplished and checks are easily encountered in experiments of that nature. One reason for this is that when vegetable tanning immediately follows the biting chrome tanning, a leather with a broken rough grain results; another reason is found in the fact that the right materials are not used for the purpose, which should be tanning substances yielding a soft tannin, *i.e.*, those which easily bring up the colour, but which do not tan with the same rapidity. As light tanning materials capable of being used for mixed tanning, japonica, cutch, gambier, sumacs, soluble quebracho extract, galls, may be quoted. Of these substances japonica and gambier are the most used in practice, sumacs very little, whilst galls have not yet found any safe use. Of all these materials it is R. Catechu or R. Gambier which has proved best for replacing mineral tan, whilst gambier, sumac and galls and soluble quebracho extracts are best applied in a complementary manner or as adjuncts to the catechu tanning. The good assimilation of the tanning matters of R. Catechu has led to the result that after having been introduced into mixed tanning and Dongola tanning, and their great use in American practice for dyed

leather and black leather, prices have risen very remarkably, thus increasing the cost of tanning. Hence the need to seek an equivalent not only in the tannery but in dyeing, where catechu preparations have now assumed greater importance and a more extensive use than in tanneries. Eitner is unaware whether these preparations advantageously take the place of catechu in dyeing, but in tanneries, as far as the substitutes tested by Eitner are concerned, catechu substitutes have not taken their place. In recent experiments on mixed tanning for coloured leather and for boot-making, Eitner observed that during the transition from the mineral tanning to the vegetable tanning certain kinds of cutch were more suitable than others; these are also the best in dyeing. These sorts are, for instance, Pegu, Coromandel, etc. They have lately been put on the market under different names and sold to consumers as catechu substitutes.

The good varieties of catechu which are employed in mixed tanning are much less used than the inferior quality, which likewise in dyeing is only slightly valued because it has not got the same tannin content as terra japonica. Eitner found that the so-called cachou, prepared by the action of bichromate on terra japonica, scarcely tans at all in actual practice, but can be used very well in mixed tanning. In Eitner's experiments on mixed tanning with catechu, his attention was drawn to a substance, which has been mentioned by others as an intermediate between a tanning and a colouring principle, and which thus promised to occupy a similar position to the different kinds of catechu and prepared catechu in dyeing, for which it appeared to Eitner to act in the fit capacity of a substitute. As a starting-point for this substitute, Eitner thought of Rhizophora barks, of which mangle bark is a species, and which are to-day offered on the market under the name of tanners' mangrove. As a tanning principle, mangle or mangrove bark has not maintained its initial price, although it has been known and "boomed" for some time. When Eitner, in 1877, gave the results of his experience of mangle, he said that in spite of its defects it might very well be used in tanning. More than twenty-five years have elapsed since its first use in tanning. Eitner was obliged, some time ago, to report unfavourably on the value of this bark, which he tested more as a dye than a tanning agent, because it had produced hollow leather, and that, even in admixture with other barks, it did not give good results, in spite of the very low cost of making the resultant leather (reasons why it could not compete). But precisely when it was believed that this tanning property attributed to the Rhizophora was not applicable in common tanning, owing to its poverty in tanning substances, its composition was found to

be of great value in mixed tanning. Moreover, just as catechu is not a current natural product, but a manufactured article, and only in that condition can accomplish its object, so Rhizophora barks are not fit to fulfil directly their part in mixed tanning without having undergone suitable preparation. Amongst the different catechu substitutes, some are to be found made from mangrove bark, in which efforts have been made to remedy the insufficiency of tanning matter by means of strengthening substances, which Eitner found useless in mixed tanning for the preparation of an appropriate product; therefore, the efforts to increase the tannin content ought to be given up and only the colouring principles contained in Rhizophora bark taken into consideration to distinguish it from real tannin extracts. Eitner received from Paul Gulden & Co., manufacturers of dye and tannin extracts, a product which appeared to him to be prepared from Rhizophora bark, which answered his requirements, and suited the object in view, *i.e.*, mixed tanning, not only as a substitute for japonica or other tannin extracts derived from catechu, but also as an ideal tannin. P. Gulden & Co. call the above product R. Catechu, and it is put on the market in both liquid and solid condition. Liquid R. Catechu yielded on testing 37 per cent. of assimilable matter. This percentage agrees with that of japonica, which is regarded as a tanning agent. Eitner avoids referring to the active principle of R. Catechu, which differs from the active principle of other tanning agents. Solid R. Catechu contains about 65 per cent. of active substances, a percentage exceeding the other better qualities of catechu. In a comparative test to determine the effects of the active substance of Pegu Catechu and R. Catechu, Eitner found them equal in the two samples: 100 parts of leather tanned by the mineral process absorbed 7·32 of Pegu Catechu, and 8·02 of R. Catechu, which gave a small difference of the skin in favour of R. Catechu. In this case, the extracts were used, in proportion to their strength, in active ingredients, *i.e.*, the Pegu Catechu with 50 per cent. of substance as 100, and the R. Catechu with 65 as 80·3, *i.e.*, 100 parts of Pegu Catechu are equivalent in working to 80·3 parts of R. Catechu. Chemists may here protest that Eitner merely took into account Pegu Catechu and R. Catechu without proving that they resemble or are equal in chemical composition, which *a priori* is not very likely, and since, like Eitner, chemists, moreover, do not know the exact chemical nature of tanning substances, they need not for the moment be discussed. The above practical results need alone engage attention, because they are of exclusive importance in industry, these two products being about equal. Taking R. Catechu for tanning purposes, Eitner assumed that the colouring principles of the Rhizophora, as well as other

vegetable colouring principles, attack the tanned fibres or fabrics mordanted with metallic salts, which does not occur at all or but little with the non-tanning substances. These colouring principles of the Rhizophora are also carried or "struck" on to the fibres of the skin, and may exercise on them a similar effect, *i.e.*, favourable for mixed tanning.

Method of Using R. Catechu.—Eitner believes that R. Catechu may be more interesting, if mention be made of the results obtained in ordinary, *i.e.*, in simple tanning. He made this test to find the independent action of simple tanning on the skin, both on sole leather and leather for belts. For the tests on sole leather, buff arseniated leather was used, because it appeared the most appropriate for tanning with a light tanning agent (the greater amount of buff leather is tanned with japonica, and it is perhaps so done now in Britain, if the price of japonica be not too high), since for buff leather for soles the red colour which R. Catechu gives is not only fashionable, but highly esteemed in Eitner's country. The tanning was carried out according to the British process, making the preparatory tanning of the skins, as they come out of the working in the river, *i.e.*, by placing them in steep, giving for the first 8° to 16° Barkometer, and for the second 16° to 32°. After this preparatory tanning, which was done rapidly and perfectly, Eitner remarked a very fine and delicate grain esteemed in the tanning of buff-hides—an astonishing thing, the more so as not having spent liquor he was obliged to use fresh juice. Instead of the three usual dips, three stronger dyes were given, because it was not desired to use any other tanning agent as a fundamental bath. The first dye was supported and strengthened for three weeks with R. Catechu, always of a strength of 35° Barkometer, the second, for three weeks, with R. Catechu of 40°, the third with R. Catechu of 45°. As, after ten days, the skins absorbed no more tanning matter, they were taken out and worked. The finished leather showed itself on the section equally tanned, was equally firm and soft, with the fashionable red colour, was very thin, but not brittle. But its yield in weight was too small, no more than 111 per cent., whilst with another tanning it was 145 to 150 per cent. It would be impossible to tan with such a result at the present time.

Eitner made a second experiment, but one of mixed tanning, with R. Catechu liquors, and afterwards with tannic agents like pine, quebracho, gall-nuts, myrobolam; the yield was 141·1 per cent. Carrying out the tanning in the fulling mill with quebracho and oak-wood extract, the yield was 139·5. The above results do not bring out the considerable advantages of R. Catechu in the manufacture of sole leather; moreover, to-day, in this manufacture, no other kinds of

catechu are used. The tanning of calf skins for leather, dyed according to the American method, has been very satisfactory. The preliminary and the final tanning are done in the same way, with known catechu liquors, only it is necessary to work with stronger liquors to produce the same effect. For japonica liquors, liquors of 18° Barkometer suffice; for catechu, to secure the same effect, liquors of 24° are required, without any superior tanning being obtained. For tanning with R. Catechu, a stronger depilatory is required. The leather obtained, according to these experiments, had all the properties of leather tanned with japonica, the reddish colour of which does not disturb the dyeing; and, as on selling, the weight is not taken into consideration, R. Catechu may then be employed in place of japonica or gambier, which costs more. R. Catechu is advantageously used in the case of white leathers with mixed treatment. To judge them better, they were prepared with japonica and nut-galls. For these tests, calf skins of an average weight of 3·6 kilogrammes (say 8 lb.) per piece were taken. After washing for two days, they were unhaired for five days in a lime depilatory, then five other days in a new, fresh and appropriate lime; they were well cleaned and placed in bran. After removing the skins from the bran, they were weighed and laid in the preliminary preparation vats. The latter was effected with a solution of 5 per cent. of common salt and 1 per cent. of alum. These substances were left to dissolve in fifty times their weight of water, and it was with this heated solution that the skins were treated in the vat for an hour. The amount of alum used here being very small, only a very thin alum tanning is obtained, which may be rather regarded as a steep, in the sense of raising the colour. But this previous steep sufficed already to bring out, in quite another way, the effect of R. Catechu. Afterwards, the skins were beaten on a block, and left to stand all night, an operation similar to the aeration of tanned goods in getting up the colour. Then commenced the vegetable tanning of the skins, which were treated in the same way. Tanning was done with R. Catechu, japonica and a fresh extract of nut-galls, or valonia, prepared cold. The first colour was given in three experiments of 6° Barkometer, and every two days the strength of the steep was increased 1°. So that the strength of the liquors on the third day was 7° and the fifth day 8°. The strength of the last liquors was, in the three parallel experiments, 15°, and the whole of the tanning was done in twenty days. In actual practice this time may be brought down to twelve to fourteen days, especially if, towards the end, the strength of the liquor is increased daily.

After this tanning the three sorts were worked together as follows. After rinsing, the skins were washed with a 1 per cent. solution

of monopol soap; they were then treated with a mixture of monopol soap and sod oil, and finally dried. The other currying was done on dyed and satin leather, tanning the first after folding with 200 grammes of sumac, and dyeing them. Once finished, the skins with R. Catechu were in each unhairing the best; one could, moreover, observe it after tanning, in such a way that, possessing the points of comparison, Eitner was entirely convinced that R. Catechu should be preferred for tanning dyed leather and leather for boots. Later on, he succeeded in imparting special properties to the leather, by modifications in the preparation of the salt and the preliminary steep. In the mixed tanning described, the alum tanning was less important than the succeeding mineral tanning. Another test had quite a contrary object, the mineral tanning with alum liquor predominated, and the vegetable only served to modify the properties of the leather. As the curriers make such leathers as "Dogskin" and "Nappa," the nappa was the object of this test. The experiments with R. Catechu on chrome-tanned leather, after final treatment, will now be described. As already mentioned, all chrome-tanned leathers, the grain of which has been specially prepared, such as boxcalf and other sorts, receive another vegetable treatment, the object of which is to retan the grain alone with vegetable matter, that being more readily applicable to a "Shagreen" finish. This retouching is somewhat difficult, for chrome tanning gives a brittle grain of poor durability, whilst vegetable tanning causes the property of the goods, as chrome-tanned leather, to disappear more or less. In experiments made with calf, horse and sheep skins, the one-steep or dip method was adopted, as being more favourable and less dangerous than the two-dip method. The following is as near as may be the method adopted. The skins are laid in a lime depilatory, twice renewed, unhaired, washed and scraped, the grain formed, and then steeped in Picol's liquor; they are then chrome tanned with Cromul A for horse-hides and calf skins, with Cromul B for sheep and goat skins (250 grammes of cromul being added per kilogramme of skin (say $\frac{1}{4}$ lb. per lb.) to the old liquor). After tanning in the pit, the skins stand for twenty-four hours; they are then drawn through Vaughan's machine, and finally folded. Skins intended to be dyed black are steeped in logwood liquor in the vat; they are then tanned with vegetable tannin, and then oiled (*tannage de graisse*). As liquor the following are the proportions per cent. by weight of the fold: 3 kilogrammes (6·6 lb.) of Licker's fat soap dissolved in 50 litres (11 gallons) of water for boxcalf and Hochlanzleder; 600 grammes (say $1\frac{1}{4}$ lb.) of potash are added, then 3 kilogrammes (6·6 lb.) of liquid R. Catechu, or 1·8 kilogrammes (4 lb.) of

solid, dissolving the latter in hot liquor; for finishing 3 kilogrammes (6·6 lb.) of bone fat and 500 grammes (1·1 lb.) of vaseline are added (Dermolin). The skins are treated with this mixture in the vat from forty minutes to an hour, according to the kind of skin: sheep and goat skins in less than forty minutes, heavy calf and horse-hides one hour. Horse-hides are treated with 4 per cent. of soap instead of 3 per cent., and the other ingredients are increased in proportion. After this treatment the leathers are left for five to six hours and dried. The dried leather is repassed, oiled with bone fat, mixed with 5 per cent. of glycerine, then redried. After drying, the leather is blackened with Carvoline B on the grain, it is afterwards finished as usual. Skins to be coloured are treated as aforesaid. After folding, they are uniformly dried. By working thus chrome-tanned leather is obtained as dry as vegetable-tanned leather; the same is the case in the dyeing, which cannot be done otherwise. The dried leathers are fulled for dyeing in hot water; the skins intended for bright colours may be prepared with 60 to 80 grammes of sumac. Washed once, they are dyed. If the leather is not to be dyed a bright colour R. Catechu may be advantageously used in place of sumac. R. Catechu greatly helps the dyeing; well-covering and well-fed shades are obtained, impossible to realise with simple chrome tanning. For the final treatment with R. Catechu, 3 per cent. on the folded weight of the leather is dissolved in 100 times its volume of water. The leather is fulled in this very weak liquor for about an hour and then placed in the dye bath. For dyeing, all the colours used for sumac-tanned leather, acid as well as basic, may be used, and there is no need to dry the dyed leather after the liquor.

Having thus obtained good results with R. Catechu, the extent of the use of this product in mixed tanning has been passed in review. This tanning process, by which Dongola leather is made, is greatly spoken of at the present time, and is used for calf and sheep skins, but chiefly for kips, horse leather for boots, ox-hides and spaltes. Very soon a great revolution will take place in the manufacture of kips, because, in the same way as in the sale of uppers, the manufacture of leather increases by mixed tanning. There is a great importation of American uppers made by this method of mixed tanning; it is therefore time that more attention was given to this method of tanning, which has the advantage of being cheap. The following are more exact experiments in this line: as raw material a low quality of C.C kips, weighing 2·5 kilogrammes, and also horse necks (*encolures*). The treatment of the kips was as follows. Stretch two days in a vat already used, but rather strong, then revat with 20 per cent. of sodium sulphide

on the weight of the raw leather. After vatting leave eight days in a lime depilatory, then four days in a fresh lime depilatory (proportion 5 per cent. of the raw leather). After washing, scraping, they are placed for two hours during the night in a bran steep (15 litres of bran per 100 kilogrammes of raw goods), then insert in a picol steep (1 per cent. of picol on the naked weight) for one hour, then chrome tan. Commence with a chrome liquor already used, of 12°; add gradually during six hours 25 per cent. of Cromul A; lay the skins on the block and leave them for twelve hours. Neutralise with 2 per cent. of the naked weight of sodium sulphide (1 of sodium sulphide in 100 of water) for two hours, wash, stretch, fold, and finally dye in R. Catechu. This final tanning is preferably done with lively colours, or failing these, with colours which may be enhanced. The first dye was given with 5° R. Catechu, which after two hours fell to 2°; it was then brought to 6°; after twenty-four hours it fell to 3°; it was again brought to 6°; after four days the tanning was finished; 30 per cent. on the naked weight of R. Catechu was used. In practice the consumption fell to 25 per cent. In the final tanning the vegetable matter very soon traverses the skins. It always penetrates in the same way, so that in each phase of the tanning process a method of comparison is at hand by which the strength of the vegetable tanning may be increased or diminished according to the properties to be imparted to the leather. A very interesting point in this tanning is that it is done with very weak liquors, and nevertheless the skins very quickly absorb the active substance; moreover, there is no need to increase the strength in the final stages, a fact which does not coincide with the rules of vegetable tanning. This peculiarity of R. Catechu renders it possible to work skins uniformly, and they can easily be submitted to the final vegetable tanning. In regard to kips they are treated similarly, and further they are washed and stretched. The increase of weight of the chromed leather was almost 4 per cent. after treatment with R. Catechu, and it could still assimilate tanning substances. Eitner, to prove this, took a portion and treated it with 20 per cent. of chestnut extract and 20 per cent. of liquid quebracho. These leathers, retanned for six hours in a liquor of 20° to 25° Barkometer, prepared as usual, gave a yield of 142 per cent. after uniform oiling. They were soft, full in the section, very uniform, very soft on the flesh side, and their colour, in spite of the chrome treatment, showed no difference with bark-tanned leather. There can therefore be produced by mixed tanning with R. Catechu a heavy, full leather of good appearance and properties, which, in spite of the tanning with chrome, is that of the bark; finally this tanning is cheap and rapid. The other kips treated only with R. Catechu were

washed, beaten and weighed. The liquor for these consisted of 3 per cent. of Licker's soap, on the already indicated weight of the leather, dissolved in 30 per cent. of water; 6 per cent. of sod oil was added afterwards to this soap solution. The skins were dipped for one hour at a temperature of 30° R. After this steeping (*liquorage*) the skins were drained and dried. Eitner working in different ways made experiments on leather of defective grain, "varnished leather," "dull sole," "boxcalf," "round grain"; they were easily made owing to the mixed process of tanning used. These results are highly important, as they enable inferior products to be used, whilst chrome tanning requires excellent products. Horse necks, ox-hides, sheep skins may be tanned very advantageously for kips by R. Catechu both as regards quality of product and yield. It is the same with varnish leathers, or for other purposes, where R. Catechu is capable of playing an important part. For all these reasons manufacturers should fix their attention on this new product called R. Catechu in Germany, or R. Gambier in Britain.

CHAPTER XI

MANUFACTURE OF LOGWOOD EXTRACT

Logwood.—Wood of *Hematoxylon Campichianum* of South America and East Indies. The chief varieties are: the cut logwood of Spain, Mexican logwood, St. Dominica or Hayti logwood, Honduras logwood, Martinique logwood, Guadeloupe logwood, Cape, etc., logwood, large logs or trunks (Laguna), or as branches and roots which are cut into chips with the cutting machine, shown in Fig. 79 and Fig. 79A, as used in the manufacture of chestnut extract. Disintegration costs a franc per 100 kilogrammes (say 5d. per cwt.). Hematin or hematoxylon is the colouring principle contained in logwood, which, by oxidation in the air, or in presence of alkalis, is transformed into hematein, which is the real colouring principle of logwood. Hematein yields coloured lakes with metallic salts, the most important of which, those of iron and chrome, are much used in dyeing black and dark colours. Alkalis turn hematein solutions from red to reddish-violet, whilst acids bring them back to yellow again; concentrated acids turn them red. The value of logwood depends on its hematin content. The following table gives the figures by L. Bruelh with different brands:—

TABLE XXXI.—SHOWING YIELD OF VARIOUS BRANDS OF LOGWOOD IN AQUEOUS EXTRACT WITH PERCENTAGE OF HEMATIN IN SAID EXTRACT

Brand.	Aqueous Extract per cent.	Hematin in Aqueous Extract per cent.	Brand.	Aqueous Extract per cent.	Hematin in Aqueous Extract per cent.
Yucatan . .	20·20	37·46	Monte Cristo	18·75	60·32
Laguna . .	21·00	47·95	Fort-Liberté.	20·30	54·11
Dominica .	14·02	53·47	Jamaican .	18·70	50·50

Drenching and Preparing the Wood.—For this operation the logwood chips should be laid in layers of 50 to 60 cm. (20 to 24 inches) thick, and so placed in sufficiently capacious space that they may be turned easily and frequently. Drenching is carried out, according to the nature of the wood and the use to which it is to be put, either with

pure water or water to which there has been added either lime, yellow prussiate, permanganate, carbonate of soda or sodium peroxide. The wood is generally sprinkled with lime water (0·5 per cent. of lime) or with a solution of potassic chlorate and oxalic acid (0·25 per cent. of each), bichromate of potash (1 per cent.), carbonate of soda (0·25 per cent.). The proportion of products employed may vary from 1 to 4 per cent. of water, and the length of the preparing process from two to ten days. The operation should be pushed to the extreme, when the wood is intended for extraction, in small autoclaves or "pears," or even when intended to be used directly in the dye vats. In that case, the preparation facilitates the development of the colouring principle by converting the hematoxylin into hematein, renders the latter more easily assimilated by the water, and enables dyers to proceed with a rapid extraction without much loss in a small quantity of water. For the same reason, the treatment of the wood under pressure, in close vessels, ought always to be preceded by rational preparation, for, in this case, it is important to work with as small a volume of water as possible, so as to obtain strong liquors, which constitute the real economical principle of the system. However, in an important factory, it would not be possible to keep wood in preparation for eight to ten days. The difficulty is obviated by the intervention of certain oxidising agents to hasten the fermentation. According to the wood, its nature, its greater or less freshness on the section, the kind of extract to be made (ordinary extract or oxidised extract, Huillard & Co.'s sort), the length of the preparation, the product or products to be used (and the proportions of the latter, which are very variable) are not at all definite, and it is the eye of the manufacturer that judges the degree of fermentation. In the manufacture by continuous decoction—in open vats—the fermentation should be moderated, because the frequent contact of the liquor with the surrounding air gives rise to a natural oxidation, which, added to a previous overdone oxidation, may lead to a superoxidation, which would cause perceptible loss of colouring principle. In such cases, a forty-eight hours' fermentation suffices. This last system of extraction is certainly much more simple, practical and economical. The manufacture, if well conducted, gives good yields, and extracts quite as strong, having at least as much body as those made under pressure. On the other hand, the latter have more freshness of tone and are more easily assimilated by textile fibres; in terms of the trade they draw more quickly.

Manufacture of Logwood Extracts.—As just seen, in a general way, logwood is extracted in open vats, the yields to be obtained being a factor of the varieties or qualities treated, the preparation which they

have undergone and the successive washings used in maceration. As in the case of oak-wood extract, logwood requires almost the same plant, except that the evaporation or concentration of the liquor is best effected in as great contact with the surrounding air as possible, so as to facilitate the conversion of the glucosides into colouring principles. To effect this the old factories, and even some existing ones, still utilise the lens apparatus, which vaporises 0·7 kilogramme of water per kilogramme of steam consumed, whereas the ordinary triple effect evaporates 18 to 20 litres of water by the consumption of 7 kilogrammes of steam or about 1 kilogramme of coal.

Yields.—These, as already mentioned, are very variable for the reasons stated; nevertheless, those got by the authors are given here, using nitrite of soda as an oxidising agent, in the proportion of 0·5 kilogramme per 100 kilogrammes of logwood chips (say ½ lb. per cwt.) and in the maceration even of a variety of Cape roots.

TABLE XXXII.—YIELD OF LOGWOOD BY EXTRACTION IN OPEN VATS

	A.	B.	C.	D.
10° extract per cent. of wood treated	74·0	55·0	...	67·5
30° „ „	24·5	18·0	20·0	22·5
Dry „ of 10° extract	18·5	17·6	...	17·8
„ „ 30° „	55·0	52·5	53·2	54·0
„ „ of wood treated	13·7	9·7	10·5	12·0
Kg. wood required to produce 100 kg. 10° extract .	135	181	166	148

A, Variety not given; B, "Aquin" logwood in logs; C, "Cayes" logwood in long logs, hard to grind, rapidly exhausted; D, Cape roots.

It is certain from the above industrial yields that the addition of oxidising agents to the maceration of the different qualities of logwood contributes greatly to an increased yield, but that ought not to prevent selection in buying certain sorts in preference to certain others, the origin of which already indicates to a certain extent the percentage of hematin. On the other hand, roots should be preferred to logs, except as regards Laguna, which up to now is one of the best qualities which reaches Havre; it is more costly accordingly. A certain sort of dry extract is found in commerce termed "resinous," which is deposited in the decantation vats, a product due to simple desiccation by the steam of the resinous matters which are deposited in the decantation vats for the 10° or 15° extract. Weak extracts deposit in winter up to 9½ per cent. (in summer only 5 per cent.), and these resinous substances are capable of yielding 10° extract once more by redissolving them in hot water and filtering, so as to keep back wood dust entrained mechanically

and other solid impurities. The following is the average composition of these dry resinous extracts :—

TABLE XXXIII.—SHOWING COMPOSITION OF RESINOUS DEPOSIT FROM LOGWOOD EXTRACT

	Per cent.
Dry soluble extract	60·0
Water	22
Insoluble	18
	100·0

Data Relating to 10° *Logwood Extract :—*

Resinous pasty extract per cent. of 10° extract	9·5
Dry resinous extract per cent.	7·7
„ „ „ „ of pasty extract	80·0
Percentage in 10° extract of resinous pasty extract	150·0

Remarks.—As mentioned above, oxidised extracts yield deep shades in dyeing, but less fast in washing, to air, light and chlorine; they have some advantages when used directly in dyeing black. Highly oxidised extracts give bad results in calico printing. In buying logwood extracts, the purchaser should be very cautious, for they are frequently adulterated. The colouring principle should always be estimated, and a dye test made with different mordants so as to assess its value.[1] The substances most often used to adulterate logwood extract are chestnut extract, molasses, dextrine, salt. Sandfort logwood contains, for instance, 50 per cent. of pure extract, 25 per cent. of molasses, 10 per cent. of dextrine, 12 per cent. of chestnut extract and 3 per cent. of salt. There is at present on the market, under the name of hematin or chrysohematin, a logwood extract of great purity. British hematin contains 95 per cent. of colouring principle (hematoxylin and hematin). American hematin contains 60 per cent., and French hematin 75 per cent. The chief French makers of logwood extract are Watrigant et Fils, of Marquettes-lez-Lille; Compagnie Française des Extraits Tannantes et Colorantes, of Havre.

[1] See "Use of Logwood in the Tannery," by H. Jossiar, *Bull. du Synd. Gén. de l'Industrie des Cuirs et Peaux*, 10th June, 1901.

CHAPTER XII

ANALYSIS OF TANNING SUBSTANCES. THE OFFICIAL METHOD OF THE INTERNATIONAL ASSOCIATION OF LEATHER CHEMISTS, WITH SUPPLEMENTARY NOTES

ONLY the official method of the International Association of Leather Chemists will be described; the processes based on the precipitation of tannin by gelatine, giving rise to errors, and the methods of Lowenthal, F. Jean or other authors being now abandoned.

Method of Analysing Tanning Substances Agreed to by the Association of Leather Chemists, London Meeting, September, 1897. I. *Sampling.* (*a*) *Liquid Extracts.*—At least 5 per cent. of the barrels should be sampled, and the sampling should be done on numbers as far apart as possible from each other. It is necessary beforehand to undo the casks selected and mix the deposit at the bottom of the cask with a suitable agitator, and to take care that the adherent deposits, whether on the sides or on the bottom of the casks, are perfectly mixed. All samples should be taken in presence of a responsible party.

(*b*) *Gambier and Pasty Extracts.*—At least 5 per cent. of the blocks should be sampled by means of a tubular tool, passing through each block in seven different places. Solid extracts should be broken and samples taken from both the interior and the surface, so as to obtain a fair average sample. In every instance, the samples should be rapidly mixed and placed immediately in a bottle or a box capable of being hermetically sealed, which is sealed and labelled.

(*c*) *Valonias, Algarobillas and other Vegetable Tanning Substances.*—It is necessary in this case to lay out 5 per cent. of the sacks on superimposed layers on a uniform surface and take several samples perpendicular to the surface. If it be not possible to work in the manner indicated, the samples may simply be taken from the centre of a sufficient number of sacks. Whilst valonia and the greater number of tanning principles may be sent ground to the chemist, it is preferable that algarobilla and divi-divi be not so sent. Bark and other substances in trusses (*bottes*) should be sampled by cutting a small portion in the middle, in 3 per cent. of the trusses, with a saw or other cutting tool. *N.B.*—If the samples be submitted to several chemists a homogeneous sample should

first of all be prepared and divided into a sufficient number of parts, at least three. Each portion is then enclosed in suitable vessels, sealed and numbered.

II. *Preparation of Samples for Analysis.* (*a*) *Extract Liquid.*—Liquid extracts should be mixed and agitated energetically before weighing. This ought to be done rapidly to avoid loss of water. Thick extracts, difficult to mix, may be heated to 50° C., then agitated and rapidly cooled before weighing. This heating should be indicated in the analytical report when it has been resorted to.

(*b*) *Solid extracts* are roughly ground, then mixed. Pasty extracts may be rapidly mixed in a mortar, and the amount taken for analysis weighed as quickly as possible to prevent loss by evaporation. In the case of non-homogeneous extracts—half-dry, half-pasty—the entire sample should be weighed, then dried at the ordinary temperature so that it can be pulverised. It is weighed once more, and the loss of weight brought out in the calculation of results as moisture.

(*c*) *Gambier.*—In the case of gambier and analogous substances, it is not possible to crush the sample or to render it perfectly homogeneous by mechanical means; therefore in this case it is permissible to dissolve the whole or the greater portion of the sample in a small known quantity of hot water, agitate to mix perfectly and weigh a portion of the solution for analysis.

(*d*) *Bark and other Solid Tanning Substances.*—The whole sample, or at least 250 grammes thereof, should be crushed in a mill until it passes completely through a sieve of five meshes per centimetre. In the case of substances containing fibrous parts which cannot be crushed to powder, the sample is first sifted, then the portion which does not pass through the sieve as well as that which has passed through it is weighed. The amount taken for analysis ought to consist of weights of the two parts proportional to the preceding weights respectively.

III. *Preparation of the Infusion.*—The concentration of the solution of tannin to be analysed should, according to the conference of 1897, be such that in evaporating 100 c.cm., the residue be 0·6 to 0·8 gramme. At the Paris Conference of 1900 it was decided that there should be taken as a basis of the amount for analysis not the weight of the dry residue, but the percentage of substances capable of being fixed by the skin, *viz.*, a solution containing 0·35 to 0·45 of matter assimilable by the skin per 100 c.cm. To secure this result it is necessary to use 30 to 50 grammes of oak bark or chestnut wood; 20 to 25 grammes of sumac; 15 to 20 grammes of valonia, myrobolams, mimosa bark; 13 to 17 grammes of divi-divi and algarobilla; 12 to 20 grammes of liquid extract; 8 to 12 grammes of solid extract.

(*a*) *Preparation of the Solution of Liquid Extracts.*—A sufficiency of the sample of extract is weighed into an open capsule or cup, and the extract run into a graduated litre flask, with about 500 c.cm. of boiling water. To do this, it will suffice to place the vessel containing the extract above a large funnel inserted in the neck of the graduated flask. The flask is made up nearly to the mark with cold water, and the flask and its contents cooled rapidly to 15° to 20° C., by a current of cold water, and the flask made up exactly to the litre mark. The contents of the flask are then filtered. According to resolutions adopted at the conference in London of 1897, and of Paris in 1900, this should be done by the aid of Schleicher and Schull's extra-strong filter paper 17 cm. in diameter, folded, No. 605. The first 200 c.cm. of the filtrate are rejected.[1]

When it is impossible to obtain a clear liquid by this process, a small quantity of kaolin, previously washed with a portion of the tannin solution, may be used. Soluble extracts are dissolved by agitation in an Erlenmeyer flask with boiling water. They are left to stand, decanted into a graduated vessel, and again exhausted with boiling water until complete solution of the soluble substances contained in the extract is effected. The sequel of operations is conducted as indicated under "Liquid Extracts".

(*b*) *Preparation of the Solution in the Case of Solid Tanning Substances.*—A certain amount of the substance is weighed, such as is capable of yielding a solution included within the limits previously indicated. The exhaustion ought to be effected at a temperature below 50° C. (122° F.), so as to yield a solution of at least 500 c.cm., then by continuing to heat to 100° C., so that the exhaust liquid contains no more tannin, all the liquors are collected and made up to a litre. If the aggregate of the solutions exceed this volume, it will suffice to concentrate the liquors from the last exhaustion. The arrangements for preparing the solutions are given farther on.

[1] It was important to define well the conditions under which filtration should be carried out. It has, in fact, been demonstrated that all filtering substances, such as paper, kaolin, sand itself, absorb variable proportions of tannin. See Searle, *Influence of the Substances Used in the Determination of Soluble Matters*, H. a. C., 1900, pp. 731 and 747. On the other hand, certain solutions of tannin remain turbid for a long time, and it is impossible to clarify them by deposition. At the Liège Congress of 1901, Dr. Paessler gave the results of experiments that filter paper No. 605 was not always uniform; in spite of that, the use of that paper was continued to the Leeds Congress of 1902, where the difficulty was to be settled. Dr. Paessler comparatively tested the use of Schleicher and Schull's filter papers No. 597 and 602, and found very notable differences in the results obtained, according to whatever number was used (*Zeit. f. Ang. Chem.*, 1900, p. 318, or *Monit. Quesn.*, 1901, p. 395 .

IV. *Analytical Determinations.* (*a*) *Total Soluble Matter.*—100 c.cm. of the clear filtered tannin solution [1] are evaporated on the water bath in a tared platinum capsule or simply in a porcelain or nickel capsule. The capsule is then dried at 100° to 105° C. in the air oven,[2] or even at a temperature lower than 100° C. *in vacuo* to constant weight, taking care to avoid loss by decrepitation of the residue. The weighed residue is calculated to per cent. of sample.

(*b*) *Determination of Non-Tannins.*—The London Conference of 1897 decided that the skin method with Procter's filter should be adopted; the decision was renewed in the Paris Congress of 1900. The arrangement adopted to carry out this determination consists of a small flask, with an elongated neck, and closed by an india-rubber stopper, through which a fine tube of 30 c.cm. in length passes, bent in the form of a siphon. The flask has a diameter of 3 cm. and a cubic content of 10 c.cm. The extremity of the tube, ending inside, is fitted with a small plug of cotton or glass wool. The flask is filled uniformly with hide powder, slightly packed, and downy. The filling of the flask, to get the best results, requires some practice; if, in fact, the skin powder be too tight, as it swells in contact with the liquid, the flow is insufficient. If, on the other hand, it be packed too loosely, the liquid runs in gushes and does not give up its tannin uniformly. The tendency, on the other hand, which the liquid possesses to pass up the sides of the flask being granted, it is preferable to pack the hide powder rather tightly against the sides, and to leave it much looser in the central part.[3] When the flask is filled the hide powder is kept in position by a piece of muslin, fixed by india-rubber.

The 1897 conference specifies that the flask should contain at least 5 grammes of hide powder; the dimensions of these must, therefore,

[1] If a balance weighing to $\frac{1}{10}$ milligramme be used, it is better to work on 25 c.cm. of liquid only.

[2] Generally three hours suffice for complete desiccation. A weighing is made after this lapse of time, then the capsule is replaced in the stove for an hour and a half; there should not be a difference of more than 0·002 gramme between the two weighings. It is quite evident that before weighing the capsule should be allowed to cool in a desiccator. If the residue be allowed to dry too long in the stove after a certain time, it will increase in weight owing to incipient oxidation even before drying is complete.

[3] In 1895, to obviate this drawback, Cerych (*Gerb.*, 1895, p. 241) proposed to prepare the filtering layer with a mixture of skin powder and filter paper pulp. He thus got over the difficulty of the tanning liquor, following, by capillarity, the sides of the vessel, instead of traversing the skin powder (see Paessler, *Zeitschf. Ang. Chem.*, 1900, p. 318, or *Monit. Quesn.*, p. 398, 1901).

be calculated.[1] The hide powder should possess sufficient absorbent power, and it should be such that, in making an experiment with distilled water, working the same way as for an analysis, the dry residue obtained by the filtration of 50 c.cm. should not exceed 5 milligrammes. The filter is placed in a Bohemian glass flask, and the tannin solution to be analysed, 100 c.cm., is gradually added to this flask, so that the skin powder is moistened by capillarity, which requires about an hour. When the filter and the flask are full of liquid, the siphon is set by aspiration by means of a small rubber tube. The liquid which flows into a graduated vessel is set aside so long as it gives a turbidity, with a clear solution of tannin; then the liquid is collected, until it gives no reaction with the first portion of the filtered liquid[2]; generally the first portion of the filtered liquid measures 30 c.cm. and the second 60 c.cm.[3] When the filtrate still contains tannin, it is better to begin the operation completely over again. The analysis may, however, be approximately corrected, by adding a small pinch of powdered skin and a little kaolin to the filtered liquid, which would in that case show a larger volume than 60 c.cm. Agitate repeatedly, let stand for an hour in a cool place, and filter through paper. When the filtration is complete, a known volume of the liquid from the second portion is drawn off, 50 c.cm. or slightly less, and evaporated on a tared capsule on the water bath and dried to constant weight in the Wiesnegg's air bath, at a temperature of 100° to 105° C., or *in vacuo* below 100° C. (see J. Noyer's drying oven at the end of this treatise). The weight found, brought to the volume of the liquid filtered, and then to 100 grammes of the substance used to prepare the liquor analysed, gives the percentage of non-tannins.

(*c*) *Determination of Moisture.*—Moisture is determined, on a portion of the sample, by drying in Wiesnegg's drying oven at a temperature of 100° to 105° C. or *in vacuo*, at a temperature below 100° C. The result is brought to 100 grammes of substance.

[1] The Turin Congress of 1904 decided that the dimensions should be 7 cm. in total length by a diameter of 3 cm., and of 1·8 cm. diameter at the neck. It was decided also that 6·5 to 7·5 grammes of powdered skin were to be taken in the future for each analysis.

[2] The soluble substances of the powdered skin, contained in the first portions of liquid filtered, constitute a more sensitive tannin reagent than gelatin.

[3] The time required for filtration, after the skin powder has been well moistened, should occupy about an hour. When filtration is very slow, and it lasts more than two or three hours, the proportion of soluble substances ceded by the powdered skin is increased, and the same occurs in the case of the proportion of non-tannins. If the laboratory temperature be too high, the weight of the non-tannins is likewise increased; it is necessary to work at 18° to 20° C.

V. *Expression of the Results.*—The results of a complete analysis ought to be expressed as follows:—

(1) *Tanning Matter Absorbed by the Skin.*—The amount of this is obtained by deducting the weight of the soluble non-tannin from the total weight of soluble substances.[1]

(2) *Soluble Non-Tanning Matter or Non-Tannins.*—Their determination has been given above.

(3) *Insoluble Matter.*—The weight of this is obtained by deducting the total soluble matter from the dry weight of 100 grammes of the sample.[2]

(4) *Moisture.*—It is calculated as above. *Remark.*—When other determinations have been made they should be given on a separate report.[3]

Supplementary Notes on the Preceding Method. I. *Apparatus used for the Exhaustion of Solid Tanning Substances.* (*a*) *Koch's Apparatus.*—This apparatus consists of a 200 c.cm. flask with a wide neck, which should be of thin glass, and well tempered to stand the heat of the water bath. This flask is closed by a rubber cork traversed by two glass tubes; the first tube enters slightly inside the flasks, the second dips down to the bottom, enlarging slightly to the shape of a funnel. The extremities of these tubes are fitted with a piece of silk gauze so as to prevent the entrainment of solid particles. The flask may be dipped into a water bath; the first tube may be put in communication by means of a tube fitted with india-rubber tubing, and a clip with a water reservoir placed 1·5 metres above (say 5 feet). The second tube, likewise fitted with an india-rubber tube and clip to form a siphon, dips into a graduated litre flask. There is first introduced into the flask a layer of about 2 cm. of fine sand washed with acids, then the known weight of pulverised tanning substances is added and the cork and its tubes inserted. There is no need to trouble about the dis-

[1] It is evident that the weight thus obtained does not represent in an absolute manner the tannin absorbed by the skin. It has been seen that the latter absorbs the colouring principles which accompany the tannin as well as gallic acid. The latter acid exists in rather large proportion in certain vegetable tans, like sumac for example. Acetic and lactic acids are likewise absorbed when they exist in the liquors submitted to analysis.

[2] At the Turin Congress, 1904, it was decided that an extract would be regarded as soluble in the cold if it did not contain more than 2 per cent. of insoluble.

[3] In certain special cases, and in particular in connection with the analysis of extracts with an alkaline reaction, it is often necessary to modify the official method in certain particular points. In such a case the analytical report should mention the method of working pursued. See *Rapports sur les travaux présentés au* 5e *Congrès tenu à Liège* (Dr. Nihoul), 1901, p. 70.

arrangement of the layers produced at this moment, because owing to its density the sand resumes its place as soon as water is introduced into the flask. Moreover, in the case of fibrous substances it is preferable to first fix the second tube after having raised the cork by gliding it along the tube; the sand is introduced and then the substance to be exhausted. When the filling is finished the cork is inserted and the whole firmly fixed or made fast either by means of string or by a special arrangement. When the water has reacted sufficiently the clips are opened, the tannic liquor from the vessel is forced into the graduated flask and replaced by pure water, the clips are closed again and the exhaustion continued. It is even preferable, to facilitate the passage of the liquor from the vessel into the flask, to add a third tube closed by a clip which is blown through after closing the first tube. The vessel can thus be completely emptied before filling it with pure water.

(*b*) *Procter's Apparatus.*—This apparatus, more simple than the preceding, consists essentially of a Bohemian vessel which may be heated on the water bath (*bain marie*); into this vessel dips a reversed funnel, the tube of which is bent twice at a right angle to form a siphon. The mouth of the funnel is closed by a piece of silk gauze and rests on the bottom of the dish. To the end of the tube a drawn-out piece of tube is joined. The bottom of the Bohemian dish is lined with sand, washed by acids, and above it are laid the tanning substances to be exhausted. Water is added and heated to the desired temperature on the water bath, and when the first exhaustion is judged sufficient the liquid is aspirated into another vessel.

(*c*) *Soxhlet's Apparatus.*—Some chemists use Soxhlet's apparatus for the extraction of tannin, but it is evident that this apparatus cannot be utilised for the application of the official method described above. In fact, in the Soxhlet, extraction is effected at 100° C., whilst, according to the preceding indications, the major portion of the tannin should be extracted first at a temperature below 50° C. The temperature of 100° C. should only be reached at the end of the process, and only kept up for as short a time as possible.

II. *Influence of the Temperature of the Extraction on the Results in Assimilable Tannin.*—It has been seen in regard to the manufacture of extracts: (1) That there existed for each tanning substance a temperature at which the extraction yields the maximum of tannin, other things being equal. (2) If the operation be performed at increasing temperatures, higher than those which have just been quoted, larger and larger amounts of tannin are destroyed, besides it is the most soluble tannins, and consequently those which are dissolved at the outset, which are

converted into inert compounds. (3) By prolonged ebullition the non-tannin extractive matter present in the vegetable tanning substances, which were originally insoluble, become soluble, and the more the ebullition is prolonged the more does the water become charged with such substances. The result evidently is that the proportion of soluble non-tannin matter is the function of the duration and the number of the exhaustions with water. From the three preceding remarks it may be concluded that the results obtained in the analysis of the same tanning substance depend greatly on the conditions under which its extraction was carried out. It was on that account that it was quite decided at the London Conference of 1897 that the exhaustion should be done at a temperature below 50° C., so as to entrain a greater part of the tannin at a somewhat low temperature, and avoid, on the one hand, the solution of insoluble substances, and on the other hand the transformation of assimilable tannin into soluble non-tannin. After this first treatment, which removes the most soluble tannins, heat may be applied at 100° C. to terminate the exhaustion, without introducing any very important errors. It is to be hoped, however, that a future conference will define, in a still more precise manner, the conditions under which this operation should be carried out, so that analyses from different sources might be comparable with each other.

III. *Preparation of Hide Powder.*—The following is the method by which hide powder is prepared at the Vienna Research Institute. An ox skin is well steeped and washed in such a way as to be well freed from blood or clot. It is limed for eight days, scraped and unhaired in the ordinary way, and finally cut into pieces of a square centimetre. These pieces are treated with a dilute solution of hydrochloric acid (1 per cent. of pure concentrated acid) until they are slightly swollen; they are washed with much water to eliminate the acid completely; stretched on a table and dried as rapidly as possible in a current of cold air. Immediately before disintegration, they are submitted for a somewhat long time to a temperature of about 40° C. (104° F.); they are then passed several times through a mill consisting essentially of two discs, the one fixed, the other mobile, the latter being fitted with teeth arranged in concentric circles alternating with the teeth of the fixed disc. The powder is first ground with the two discs a little apart, the operation being finished by bringing them much nearer. The method just described is almost identical to that given by Schroeder;[1] however, in the latter, drying is carried out at a higher temperature, and moreover a steel mill is used identical in shape

[1] *Berichte der Commission zur Feststellung einer einheitlichen methode der Gerbstoffbestimmung* (Cassel, 1885).

with a coffee mill. It may finally be pointed out that the skin powder of Freiberg contains variable proportions of paper powder. Several

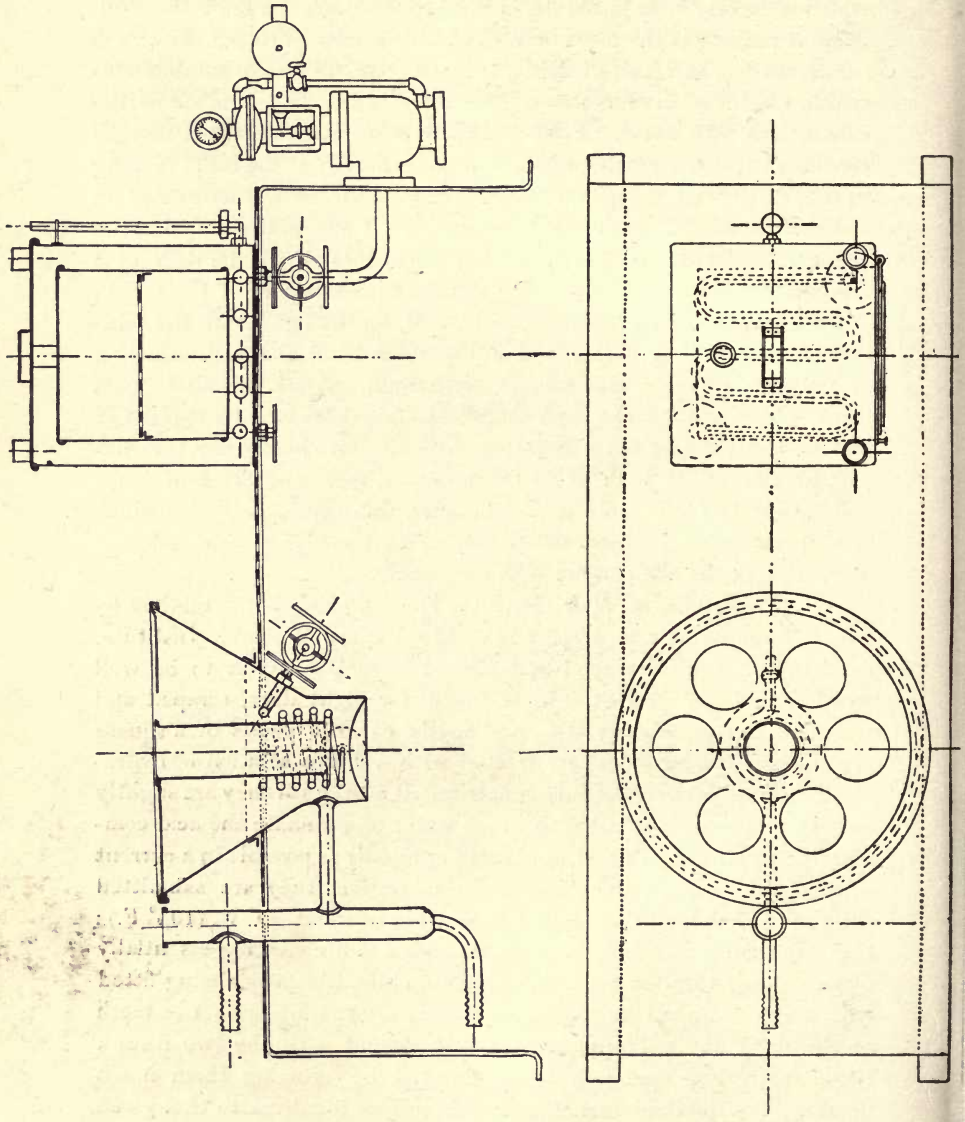

FIG. 103.—Water bath for the estimation of soluble matter and non-tannins in the analysis of tanning substances (J. Noyer).

chemists, Procter in particular, have made comparative tests of different species of skin powder, but the results obtained up to now

are not sufficiently conclusive for there being occasion to use any one determined skin powder exclusively.[1]

The conditions which a hide powder intended to fill Procter's filter should fulfil are the following:—[2]

(1) It should be white, soft to the touch and downy. If it possess a putrid odour and a greyish colour it should be rejected, because it has been derived from a skin which has undergone putrefaction, and the results obtained by its use are abnormal.

(2) In making a blank analysis with distilled water, rejecting the first 30 c.cm. and evaporating afterwards 50 c.cm. of the filtrate to dryness, a greater residue than 4 to 5 milligrammes should not be obtained.

[1] At the Leeds Congress, 1901, it was provisionally agreed that Freiberg skin powder, containing as a maximum 20 per cent. of paper cellulose (Mehner and Stransky, Freiberg, Saxony) should be used for the application of the official method. At the Turin Congress of 1904, this resolution was confirmed up to the Frankfort-on-Maine Congress, September, 1906.

[2] Procter, *Leather Industries Pocket Book*, 1898, p. 115.

APPENDIX TO CHAPTER III., PART I

The continuous rectification plant for wood-spirit, described on pages 58-62, does not include a description of Guillaume's continuous rectification plant. The following description and claims in respect of Guillaume's method, kindly supplied by MM. Egrot and Grange, of Paris, speak for themselves.

Continuous Rectifier for Rectifying Wood-Spirit

The continuous rectifier, on Guillaume's system, shown in Fig. 104, used for wood-spirit, is a modification of that used for ethylic alcohol (grain, etc., alcohol). In this apparatus the products are separated in a regular manner. The difficulty of separating the pure products from the impurities will be appreciated, from the fact that it is necessary to vary the quantities extracted precisely according to the impurities present in the liquids to be rectified. The efforts of Guillaume have been directed towards obtaining the necessary regularity by fitting the apparatus with a "fly-wheel" to cope with slight fluctuations. The following description explains how Guillaume has secured the remarkable results to which the adoption of this apparatus is due. The plant, as a whole, consists of a purifying column BB', in which the most volatile portions are removed; the alcohol then passes into the concentration column, C, which is supported by a wrought-iron receiver, V, called the accumulating receiver. The last runnings are extracted in the lower part of the concentration column and the almost pure alcohol is extracted from the top of the rectification column to undergo afterwards a slight distillation in the column DD', in which the final purification is accomplished, from which the alcohol passes to the exit test glasses. The spent or exhausted phlegm issues from the concentration column to pass into the exhaustion column, E, from which it is ejected quite free from alcohol. The plates on which the concentration of the vapours and their purifica-

tion are accomplished are of a peculiar system in which the liquid is made to undergo a bubbling action as many times as there are caps. These plates have considerable exhaustion capacity. The accumulating receiver (Fig. 105) mentioned above plays an important part in the working of this apparatus. According to Guillaume, amongst the ob-

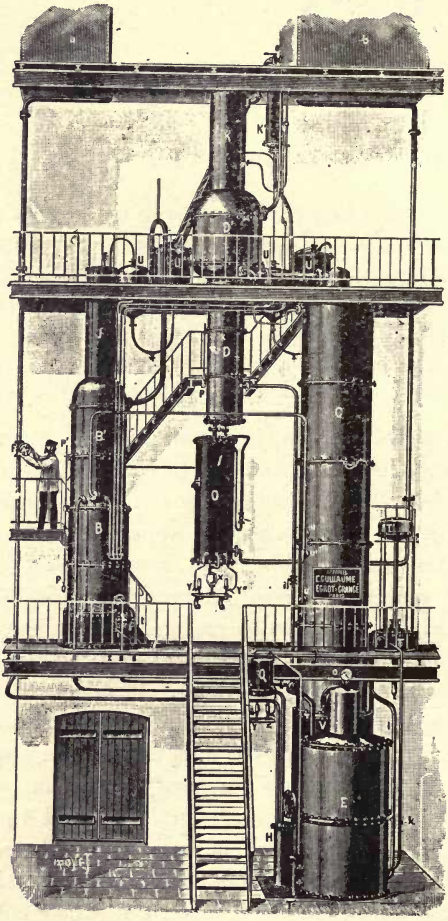

FIG. 104.—Continuous rectifier for rectifying wood-spirit.

stacles to continuous rectification which his predecessors encountered, the most important was the regulation of the apparatus, into which on the one hand there enters an alcoholic liquid, and from which on the other hand there issue (1) the liquid from which the alcohol has been extracted, (2) the alcohol which has been separated from its im-

purities and collected apart. It will be readily understood how difficult it must be to adjust the apparatus, so that the pure alcohol and the impure alcohol extracted represent very exactly the same quantity as that which entered therein. The delicate point is to get the alcohol issuing from the columns by the test-glass taps to balance with the alcohol introduced by the feed, without which balance the strength lowers at the *bon goût* (purified alcohol) test-glass tap, or, on the other hand, a portion of the alcohol goes down the drain with the residual waters, if matters be not remedied in time. The permanent supervision of the working of the rectifier does not remedy

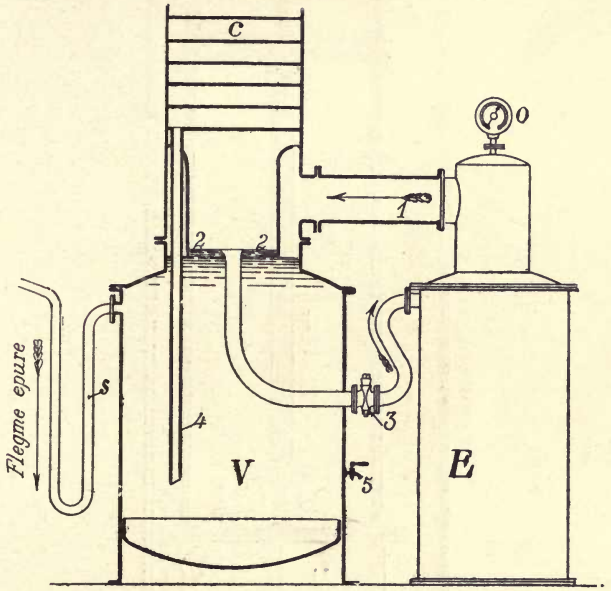

FIG. 105.—Accumulating receiver.

these serious drawbacks, but in an imperfect manner. Whatever efforts be exerted, variations in the strength and purity of the rectified alcohol and loss in the spent wash cannot be avoided. The use of the accumulating receiver deals with these difficulties. This consists of a receiver, V, constantly traversed by the alcoholic liquid at a certain part of the apparatus, and is completely filled by this liquid. It forms a sort of regulating "fly-wheel" of constant volume, capable, owing to its mass, of automatically absorbing the momentary excess of alcohol which may be produced in the column without the general working being affected, and in such a way as to place the routine working of the column under stable equilibrium. At the spot where

this accumulating receiver acts the alcoholic liquid may very well lower or increase in strength by 10° or even 20° G.L., without that

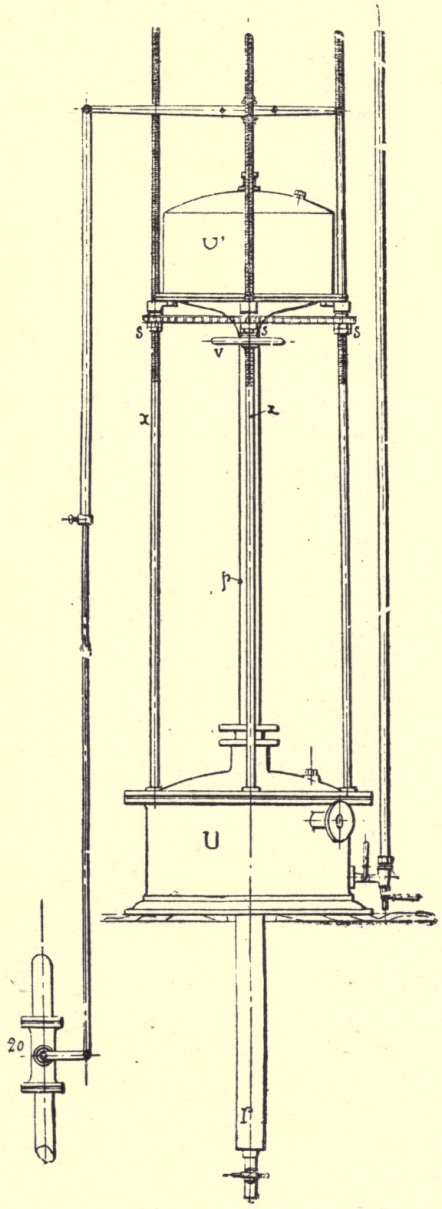

Fig. 106.—Steam regulator.

causing any perceptible repercussion, neither on the plates above nor on the exhaustion, and without any appreciable disturbance of the classification on the plates. By giving to this receiver, for instance, a capacity of 20 hectolitres it will absorb or restore 4 hectolitres of 100 per cent. alcohol for a total variation of 20° G.L. on the plate, say, 10° G.L. below or above the average degree. It will thus easily cope for four hours with either insufficient or excessive feeding, corresponding to 100 litres of 100 per cent. alcohol per hour on the flow from the test glass. This example will suffice to explain the important function which is attributed to this organ in Guillaume's apparatus. The regularity of the apparatus is still further secured by means of the

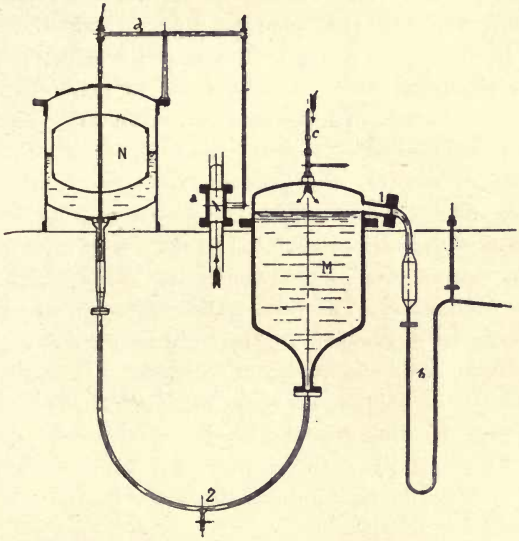

FIG. 107.—Automatic water-feed regulator.

automatic regulation of the steam and the cooling in the condensers. The regulation of the steam heating is accomplished by an adjustable regulator (*regulateur à régime variable*) (Fig. 106), by which the working pressure may be varied at any moment without stopping the working of the rectifier. The consumption of steam is greater in any given apparatus the greater the pressure at the bottom of the column ; that is to say, if this pressure be increased the consumption will be greater, and, on the contrary, it will diminish if the pressure be diminished. The advantage of being easily able to regulate this working pressure so as to adapt it to the work to be done by the apparatus and to keep it as low as possible will be readily understood. If the amount of liquid to be distilled increases, the pressure is increased so that sufficient dis-

tillation may be produced whilst thorough exhaustion is at the same time maintained. If, on the other hand, the quantity of liquid to be distilled diminishes, this pressure is lowered so as only to consume the steam required to ensure thorough exhaustion. Fig. 106 shows this regulator in which the upper vessel is supported by screw bolts forming pinions which can be wrought by means of a chain. By turning the pulley, V, these screw bolts are caused to ascend or descend together, and consequently also the upper receiver, which thus increases or diminishes at will the height comprised between the upper and lower receiver. The vertical rod which manipulates the steam distribution valve is extensible so as to adapt it to the variation in height of the upper receiver. Finally the bottom of the lower receiver has a sheath containing the plunger pipe (which slides therein) through which the liquid going to the upper receiver passes. The water feed is regulated automatically, so as to maintain a constant pressure in the condenser. Guillaume's automatic water-feed regulator consists of two communicating vessels (Fig. 107), of which one, which is completely closed, receives the pressure from the condenser. This pressure acts on the water contained in the communicating vessels ; and, under its influence, the water rises into the other receiver which is open to the air, and raises a float, which works the valve which feeds the water into the condenser. The value of this appliance on a very simple plan is more especially enhanced by the right proportion of its organs, its good construction, its absolutely safe working. It regulates within very narrow limits the amount of water with which to feed the condenser according to the vapours to be condensed. Guillaume's apparatus works, generally, with the water tap full on. It is the valve of the regulator which alone regulates the water feed.

THE END

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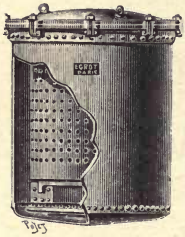

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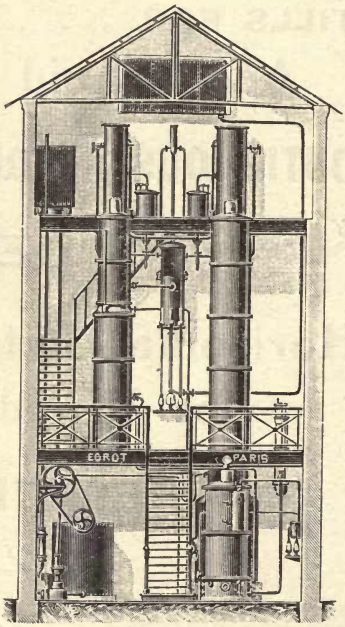

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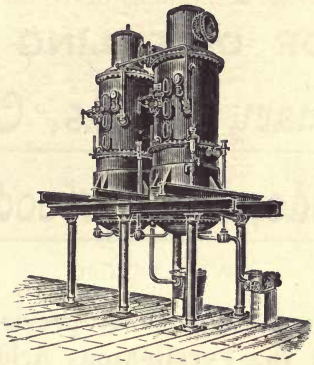

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